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# The Modern Dyehouse

**The Superintendent and His Specialized Staff—Striving for Greater Efficiency by Control—Making the "Lab" More Than a Luxury—Easing the Chemist's Burden with System—Reducing Matching to a Science—The Human Element**

By ORMOND W. CLARK

THE modern dyehouse, in line with the trend of scientific management, should distribute its activities under the supervision of specialists. The technical phases of dyeing are too complex, the intensive production requirements are too exacting, and the labor problems are too profound, to permit the efficient execution of all their details by one individual.

To meet the new conditions, the functions of the dyer must be expanded, and the resultant functional divisions must be separated and placed under the supervision of men especially qualified by training and experience to develop them with the utmost efficiency. To co-ordinate these functional divisions with each other and with the other departments of the mill there is created the position of superintendent of dyeing.

The superintendent assumes the executive functions of the dyer, but delegates the routine functions to specialists. He should be a high-grade executive, technically trained in chemistry and engineering, with sufficient practical dyehouse experience to mellow his judgment and to guide his theory into profitable channels. He is responsible to the management for the maintenance of quality standards, production schedules, minimum costs and technical superiority. He must co-ordinate dyehouse, drugroom, laboratory and plant to achieve the minimum production cost consistent with a high quality output.

Untrammeled by routine production details, the superintendent is free to investigate dyehouse procedures and to determine the optimum dyeing conditions with great exactitude. The old-time dyer, busy, and worried by countless details, found it difficult enough to develop formulas that would work, without undertaking the careful experimentation required to find the *best* formula.

To aid in the analysis and improvement of dyehouse procedures, the superintendent can marshall the scientific knowledge and precise technique of the laboratory. From a search of the literature and by visits to other plants, he can determine the best contemporary practice.

Better dyeings at lower cost will result. Full utilization of raw materials will ensue. Machines and men will exhibit their maximum productivity. Simplification and standardization will increase dyehouse efficiency.

## Striving for Greater Efficiency by Control

Dyeing machinery is minutely analyzed to discover possible improvements, which will improve the quality of product, raise the permissible operating speed, or require less labor exertion. Minor changes in machine design often greatly increase operating efficiency. Whenever possible, fallible human control is replaced by exact mechanical devices, temperature controls, level regulators, or chemical tests. Dissection of dyeing processes into elements, studying the *raison d'etre* of these elements, the justification for each step in the light of the best scientific knowledge, and postulating the possible improvements, often permits increased efficiency. Possible profitable variations in dyehouse procedure are submitted to careful laboratory trial, then to small scale plant trial, and finally to full scale plant trial.

The superintendent of dyeing examines all orders for dyed goods before they enter processing and designates the preferable treatment best to prepare them for dyeing. Thus adequate preparation is secured, but unnecessarily involved or expensive preparation is prevented.

Through frequent conference with the plant salesmen, information is secured regarding customer opinions, competitive products, and advanced styles. Suggestions of novelty effects and improved products are made with the hope of attracting new business. The salesmen gain sufficient familiarity with dyehouse re-

quirements and possibilities to considerably enhance their selling intelligence. Customers' criticisms are ascertained at their first inception.

Frequent conferences with the management keep the superintendent of dyeing fully informed of the many indirect factors bearing on his work. Market trends are anticipated and drugroom stocks are kept at a minimum. Discussions of new fabric constructions show the feasibility of handling them on available equipment. New shades receive a preliminary estimate of dyeing cost.

Continual check is maintained upon all the details of dyehouse operation. The matcher, the floor men and the operatives are kept on their toes. The matcher's work is checked for accuracy of match, correct proportioning of liquor to material, number of adds per shade, and total output. The floor men are checked for machine operating time, and for their general thoroughness of floor supervision. Equipment condition, steam supply, water supply, and other mechanical conditions are periodically scrutinized so that repairs and delays are obviated before they affect production.

### Making the "Lab" More Than a Luxury

A most valuable aid to the dyehouse is a well staffed and well equipped laboratory, organized as a profit-producing unit, and not maintained by the mill merely as a costly luxury or as a necessary evil. A properly supported laboratory soon proves to be a distinct asset to a mill and especially to a dyehouse.

The most obvious function of a laboratory is the assurance by analysis that all raw material shipments conform to the set standard of strength and purity and that their money value is not surpassed by competitive products. Any textile chemist can testify that it is not unusual for a plant which has not previously employed a chemist to be buying highly adulterated dyes or mixed dyes at an exorbitant price. This situation is not unique with the dye field, but extends to other raw materials, especially to finishing compounds and to scouring assistants. Even when all dyes and raw materials are purchased from reputable dealers who know that deliveries will be checked by analysis, an occasional variation in shade or strength will appear. Issuance of forewarnings to the operating departments prevents costly damages and delays to actual production.

As essential as the testing of raw material is the checking for fiber type and dyeing properties of all goods entering production. Canton, Italian, tussah, viscose, nitro, acetate, and other real and artificial silks; linen, rhea, jute and such fibers; and the fibers with chemically changed properties, of which immunized cotton is an example—all must be differentiated before production dyeing of a lot is undertaken. Among the artificial fibers especially, a difference in dyeing properties sometimes occurs from lot to lot. It is worth-while to test every lot of even such a staple fabric as cotton, to insure its conformity to the standard reactions established for that particular construction and manufacture of fabric. To designate the characteristics and peculiarities of goods by simple laboratory tests before production dyeing, is quick and inexpensive; to observe them first during plant processing is disconcerting and may be expensive.

To maintain a high standard of quality, the laboratory should check the dyehouse output from time to time. Penetration and levelness are observed. Fastness to the destroying influences of light, washing, perspiration, and other similar agents is carefully compared with the established standards. For guaranteed fast color work routine fastness tests are advisable on all lots.

Technical superiority is promoted by the perusal of contemporary periodical literature, native and foreign. Tests and analysis of competitive products secured by the salesmen discover any original advances in the art and may permit duplication or improvement. Close contact with the dye manufacturers insures up-to-date files of color cards and dye samples. This is about as far as most of the plants now go to maintain progress. Research, with rare exceptions, is still too altruistic a conception to be actively fostered by the American textile industry.

### Easing the Chemist's Burden With System

In the majority of plants the chemist is too busy with the multitudinous exactions of general plant control and with the never-ending stream of troubles flowing in from the production departments, to devote a full measure of justice to the dyehouse. As the complex conditions arising from new fibers and complicated dyeing processes continually increase, the load becomes more onerous to the already overburdened chemist. The superintendent of dyeing possessing a sound technical training, eases the burden upon the chemist and facilitates the smooth co-ordination of laboratory and dyehouse.

To promote accuracy in the dyeing formulas and to provide the data for a good cost system, there is an orderly, well systematized drugroom under the joint supervision of the dyehouse superintendent and the plant chemist. Powdered dyes are kept in tight, self-closing, easily accessible containers. Pastes are stored in containers which minimize evaporation and permit rapid, thorough agitation before withdrawals. Chemicals are kept in dry, substantial containers with a cover. The stock is maintained under the proper conditions of temperature and humidity to insure optimum preservation. Powders and dry chemicals are kept warm and dry. Pastes are kept cool, away from air currents, and are never exposed to freezing tem-

(*Continued on page* 168)

# Proceedings of the American Association of Textile Chemists and Colorists

## The American Association of Textile Chemists and Colorists

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## JANUARY MEETING OF THE RHODE ISLAND SECTION

The January meeting of the Rhode Island Section of the American Association of Textile Chemists and Colorists was held on Friday evening, January 6, in the rooms of the Providence Engineering Society, 44 Washington Street, Providence, R. I.

The following officers were elected for the year 1928: John Hutton, chairman; Albro N. Dana, secretary; Ben Verity, treasurer. Sectional Committee—John G. Masson, Karl R. Moore, W. H. Wingate, Herbert F. Schwartz.

The Nominating Committee consisted of the following: Alfred F. Lustig, Allison R. Fletcher, Ralph F. Culver.

Daniel R. Weedon, of the Hungerford & Terry Inc., Clayton, N. J., presented an address on the subject, "Purification of Waters for Industrial Purposes." This address follows.

Forty-eight members of the Section were present.

A. N. DANA, *Secretary.*

## Purification of Water for Industrial Purposes

By DANIEL N. WEEDON
*Hungerford & Terry, Inc.*

Mr. President and Gentlemen: In talking to you about filtration, I am talking to a number of men I already know and to whom I have already spoken on this subject. But I want to say, in starting out, that so far as a textile plant is concerned, there is practically no water that is impossible. If you have the laying out or placing of a textile plant, or any other industrial plant, and other things that come into consideration are satisfactory or advantageous, the water problem doesn't need to affect your decision very much, because for all practical purposes in dyeing or bleaching of silks or woolens or cottons, any dirty water, practically sewage, can be made entirely satisfactory at a very reasonable expense. I say that because of what I have seen done, and a little later this subject might interest you enough to bring out, by questions that you will ask me, the things you are interested in. Otherwise I won't know. I merely will cover points that I have outlined in advance.

The first thing in examining any water supply is to know the purposes for which you intend to use it, and coupled with that the quantity you will use, and very

closely, the elements of a foreign nature that are in the water and which of these elements are objectionable.

### Turbidity and Color

Those are the first things to consider. In general, the factor of most importance is *turbidity*, because that must be taken out. Even a woolen mill doing scouring or very heavy coloring, ought to have turbidity removed. Then follows *colors,* which are closely akin to turbidity; that is, any suspended matter that sand filtration could remove. Color might be made up of the same volume and plain sand filtration will not take it out.

Water might also contain alkali, nitrate, calcium, magnesium, iron and possibly odor and taste. Inasmuch as I am talking about water for industrial purposes only, taste does not come in. Odor must be considered. Some waters in New England, after proper filtration and after standing for a reasonable number of hours, will begin to smell. Just why that has not been taken out, I don't know. It can be killed by copper sulphate, but that is also objectionable.

Take water with a turbidity of any degree varying from 5 up to 100, and in Southern waters 3,000 or 4,000; that is, in parts per million. That copper can be removed by plain sand filtration. Color must be chemically treated and the usual method, as many of you know, is to run the water by gravity or pumping into a sedimentation basin, allowing it to stand, varying with the type of water, anywhere from two to four hours—an average of three hours in this basin, treated as it goes in with a chemical to coagulate the color. That chemical is alum. To make alum effective it must be combined with enough alkali so that the alkali will combine with sulphate of alumina, which is what coagulates the color into particles large enough so that the sand bed can hold them up. Some colors can be taken out without chemical treatment. This feeding-in of alum would depend upon the water itself and must be worked out in each individual case very carefully in the laboratory. I should say you might need to feed in 1 to 1½ grains of alum per gallon, and the amount of alkali which you would feed in would depend on the natural alkali of the water. If 7½ parts soda-ash is increasing your alkalinity to 17, you want the original alkalinity to be 8 or 10, or possibly more after filtration, therefore you should increase or decrease alkalinity in order that the result will be 10 or more. Recently sodium aluminate has been used instead of alum soda ash and we have made some very desirable examinations; if you want any information on that later, I will try to answer questions.

Alkali can be removed by alum and copper sulphate. Calcium magnesium causes a hardness and that means a softening job. Iron is an alien substance, treated by coagulation and is fairly complicated. It is the most expensive substance to remove. It is usually accompanied by a hardening of the water. The more things there are in the water, the better I am pleased—from the filtering standpoint.

### Types of Apparatus Used

We will now discuss the type of filter to be used. There are the gravity and the pressure types. It is impossible to recommend a type for your plant when you say it uses 100,000 gallons or a quarter of a million gallons. The result must be worked out from a water analysis. Where the equipment can be run by gravity to the plant, a gravity filter might be best. If it has to be pumped directly in, a pressure filter might be better. In general a gravity filter runs at a slower rate because you have the big tank of water to force through the pressure filter. It can run either without or with a sedimentation basin. For a small plant—and by small I mean 1,000 or 2,000 gallons per day and no sedimentation—where you merely want to take out the turbidity, I would say, use a pressure filter. On the other hand, if you are going to use 10,000 or 15,000 gallons and where it requires two or three hours sedimentation, it would probably be better to let it run to a gravity filter and from there to the plant, even if it required a second pumping. The pressure filter is only vertical. The gravity filter is both vertical and horizontal. In fact, right here within the limits of this city we have a pressure of 400,000 gallons per minute, which is quite a little water, and they are using a pressure type. For a gravity filter the basin can be either wood, concrete or stone; usually they are concrete or wood.

I have here a picture showing one type of gravity filter and I am going to refer to that now. As to the filter construction, if you are interested in that, I think it would be about the same so far as the strainer system—the sand and gravel—is concerned. This shows a conventional feature of a wash rate type of filter. What I want to show you is this wooden tank here [indicating]. That could also be made of concrete. It can be concrete with this type of washing. This [indicating] controls the raw water coming into the top of the bed. It is then filtered through the sand. A strainer system is placed in the bottom made of pipe—literally nozzles—for distributing water underneath the bed and designed for as much permananence as possible. When this is filled in with stone and gravel, you don't want to empty it out very quickly. The layer of stone is for distributing the water under-

neath the bed on your back-washing and to give the sand a thorough scouring. The most perfect back-wash you can get is none too good. The stone decreases in size up to a line of gravel large enough to stay on top of this bed of stone and support a bed of sand about 3½ feet deep. This is simply a division bed.

The sand must be carefully selected. I will say that, in general—and this is only approximate—an average filter sand would probably give you about 400 grains to the square inch. The diameter of sand for a given water might vary from 35 mm. to 60. A properly designed and laid-out filter must take that into consideration, and anybody putting in a filter cannot give too much attention to the size of the sand and uniformity coefficient. That is the last barrier between a dirty, raw water and your valuable goods in a finishing plant.

### Procedure of Washing the Filter

Now, I said that the strainer system and this stone are to diffuse the water for back-washing. The washing of a filter is one of the most important things that happen in the filter plant. On a back-wash it is necessary co scour the layer of sand as though you were churning it. The theory of filtration, as we interpret it, is different from what some of you men in this room may interpret it as. We say that it involves the entanglement of this coagulated coloring matter in the bed of sand; it is the matter of the dirty sand right on top, which might be termed "surface straining." We might expect it gradually to work down through the sand; therefore all grains must be very thoroughly scoured.

We put in the bottom a given amount of water, which is approximately 9 gallons to the square foot of area. That comes up to the sand. This wash rod is revolved around through the sand for five or six or eight minutes. That [indicating] carries the water out into a water pipe and away. That is merely one method of scouring sand. You can also introduce another system on the gravel. That consists of heavy brass piping; and here [indicating] it is connected with the radiator system. The same volume of water comes up through the sand and gravel and is joined here [indicating] by two-thirds of a cubic inch of air, and that air gives the sand a very thorough turning over. The advantage of mixing it in here [indicating] instead of here [indicating] is to prevent the mixing of the gravel in this area with the sand, gradually working the gravel toward the top and the sand down, eventually putting your filter out of commission. Whether stirring with the water and the rod or with the combined water and air, the purpose is the same, being to scour that sand thoroughly.

Now, in this coagulation of coloring matter, which is caused by your alum and ash or sodium aluminate potash, you are introducing in your water something that can be very troublesome if you are not careful. That is, alum. In the first place, alum is going to cause complaint in every department, from the boiler room straight through the plant. But the filter must get alum through in some form or other. Another reason is that alum is expensive, and bringing water to the proper point with either acid or alkali is cheaper than with alum.

In general, the filtration is slower on a gravity filter than by pressure filter. The pressure filter can put through 200 gallons per minute. This can be exceeded quite materially without trouble.

I am going to skip over for a moment the chemical treatment. The next thing I want to speak of is the control of coagulation. We find that hydrogen ion concentration is the simplest and probably the most effective way of controlling what we call coagulation. We can do other things, but a pH test is very simply done. Any workman can do it after a few demonstrations, and if you show him where you want the pH, or where you want the sedimentation basin held, he can hold it there very easily and accurately. The pH range is usually from about 5.8 to—well, 6.6, which, as you see, is on the acid side of the sedimentation. That, of course, means right away that you ought to get proper coagulation in this area of the square; and we pass that water through to the filter water reservoir and we have given you an acid water. That is very simply corrected; for since you bring it up to 7.2, it is non-corrosive. That is easily accomplished by feeding in a little sal soda, a very inexpensive chemical.

### Control of Coagulation

I do not want you to hold me to a statement that proper coagulation of water must be between 5.8 and 6.6—within a narrow range right there [indicating]—because there are waters, and extraordinary waters, where coagulation will take place to the best advantage as low as 5, and in a few cases as high as 7; but those cases below 5.8 and above 6.8 are exceptional and should be considered as such. We can come back to that pH discussion and I shall be very glad to answer any questions if you have any; and, in fact, we can run some pH tests for you. We have all the equipment here.

The test for turbidity is very simple. The test for color is based on the comparison of standards which were developed by Hazen. They are mostly based on

accepted set standards. The test for pH is, as I have said, simple; and if any of you want a demonstration of that we will be glad to make it for you. The test for alum is a standard hematoxaline test and is extraordinarily accurate. I don't think any of you would worry very much if you had an alum part of one-quarter point per million and the restorative alum test would show one-tenth part per million. With a little practice you could show closer than that. I would be very glad to demonstrate that point. Alum can come in filtered water. Sulphate of aluminum can come through. That would probably be a case of too much alum. You might pass alum hydrate through. The alum test would show it, but would not show which it was.

Another objectionable thing from several standpoints is your $CO_2$. Rust is one objection, and the effect on your dyes is another. Interference with coagulation is another. It can be very greatly reduced, in the mixing in of your chemicals, by proper agitation. It is impossible for me to stand here and tell you the limits of $CO_2$. In some cases low $CO_2$ is advisable. A twentieth part per million in many cases might be very objectionable.

In New York State, with water of a high alkalinity, the water might have 100 parts per million of $CO_2$ and have no rust. There is no hypothetical case that I can point out as too high.

I shall have to return now to the two things that I skipped earlier in the evening. First, removing calcium magnesium hardness. That is a job for a zeolite softener. A softener has almost, but not quite, the same construction as a filter. Usually a softener is a pressure equipment rather than a gravity type, but not always. We will assume that the bed of zeolite is put in, in about the same form as in the filter. In the discussion of softeners many people still have in mind the type of zeolite used some years ago. That was synthetic zeolite and very unsatisfactory. Its exchange value was low; that is, its properties of absorbing hardness was low. It took a long time to regenerate. Its life was very short, and the pores would fill up with dirt from the water and it wasn't at all successful. I don't think I should use the word "unsuccessful," because they did soften water, but it was not entirely satisfactory.

Then natural zeolite was discovered, which is merely a grain-edged sand deposit, the two best beds being in New Jersey. You take the zeolite grains out of the ground, wash them and harden them for grains of sand—possibly a little larger than the grains of sand in the filter—and this sand has the very valuable property of absorbing 3,000 grains of hardness per cubic foot of zeolite. If you want to soften a certain amount of water of 1 grain hardness and you have a certain amount of time to do it in, you must figure the softness on two bases. One is the amount of water that the softener will pass through per minute—that will probably be in the neighborhood of 2 or 3 gallons per minute. As soon as the zeolite has absorbed from the water so much hardness, the zeolite is totally exhausted and the water will pass right on through. All that is necessary is to give the zeolite a good dose of brine, the amount being about a half pound of salt for each 1,000 gallons. That effects the regeneration. With the old synthetic zeolite it took eight or nine hours. Now it takes thirty minutes. It is softened in the morning and will absorb from morning until noon. During the noon hour it should be regenerated and it will work all afternoon, and then it should be regenerated again at night. It is a very simple process, because this zeolite simply takes from the water chloride of magnesium and sodium. Salt replaces them and washes them out, and they are ready to start again. The life of zeolite is as long as the sand in the filter. It is not quite as hard, but it will stand the abuse of filtration and softening, and will hold up apparently indefinitely. I shouldn't quite say that, because we haven't known natural zeolite long enough. The exchange value, however, does not seem to drop.

I am going to say that so far as dirty water is concerned, it affects every process in your mill many times more than you realize—all the way from the water plant to your bleaching house, and then on to the dyehouse. In fact, I can say in general the only variable product in your plant is your water supply. Your cloth almost always comes without variation. Your dyes are quite accurately made up before they come to you. Everything has a fixed value except your water supply. Those of you who have raw water to deal with realize that variation in turbidity can range many times within a few days from 15 or 20 to several hundred. Color can vary in almost any stream from a normal 20 or 30 up to 150 or more. That is a very ordinary range. Hardness will vary in many streams from 2 to 8 or 10 grains. The iron will also vary considerably. But all these things can be corrected.

### Estimating Costs

How you chemists and dyers can balance your chemical formulas, when formulas must be so exact and when water varies so very widely, is beyond me. The cost of getting good water is really very low. If you asked me how much a filtration plant for a million gallons a day would cost I couldn't tell you; I am not here to do that, anyway; but it depends upon just what you want. Let us say that, in general, a woolen

mill would not want to reduce the color lower than possibly 20 or 25; or sometimes the color is satisfactory but they do want to eliminate turbidity. $CO_2$ might or might not be present. Therefore it is very difficult to tell you what it would cost you in your plant to give you clear water from your river. The problem is as varied as the kinds of water.

In New England, I can say, it is very probable that there is no filtration plant, not even a city plant, whose cost per 100,000 gallons is as high as $5. That is, taking water from any stream and giving it to you absolutely crystal clear: zero turbidity and five in color. I doubt if there is a plant as high as $5—$3 might be an average—and when only taking out turbidity it would be 50 cents per 100,000 gallons; that is, on the basis of 100,000 gallons. The cost of softening is lower, on the average, than the cost of filtration. You should bear in mind that you cannot put dirty water into a softener; it would so foul your zeolite grains that it would put them out of commission very quickly. Assuming that the water is clean already, you can soften water; and in order to have some basis for my figures, I have taken 100,000 gallons of water with 1 grain of hardness. Seventeen parts per million, however, would take in, I think, most of the streams in this vicinity. And the cost of softening that water would be under $1 per 100,000 gallons. It might be as low as 50 cents. When I mention these costs I mean all the expenses, including interest, depreciation and perhaps a single pumping and labor—that all goes with it—and the chemicals. If there is necessarily a second pumping, you might have to add in the neighborhood of $1.50 for that second pumping.

I have more or less skipped through this subject of filtering and softening, trying to give you, in the first place, the idea that there isn't any water we cannot give you perfectly clear and perfectly soft, continuously. I have tried to picture different types of filters and what we try to accomplish in filtering and softening, and have touched very briefly on the methods of determining and controlling the amounts of chemicals fed into your water, which is almost universally done on a basis of pH value; and have also indicated a test for pH in filtered water and for the alum content. You know what particular points of filtration you are interested in more than I do, and I think rather than for me to go ahead with a list of minor topics I have selected and just simply hand them to you, I would very much prefer to give you something for your courtesy in coming here, and to give you that I think you had better tell me what you want to know. I will be very glad to elaborate on any point or demonstrate some of the chemical controls. I think that would be the better way to give the information you want.

## Discussion of Water Purification

*Mr. Norman*—Mr. Weedon, can you tell us how long it should take to rewash the sand in filters?

*Mr. Weedon*—In a filter properly designed it should take—even then the time varying on the amount of dirt in the sand—from five to eight minutes.

*Mr. Norman*—I find that everyone varies on that question. They say five to eight minutes; but when my men have cleaned it they tell me it took forty-five minutes to an hour, and I find they are right. It takes our filter now at least forty-five minutes to wash it.

*Mr. Weedon*—Is that a pressure or gravity filter?

*Mr. Norman*—Pressure.

*Mr. Weedon*—If it takes forty-five minutes it means that the filter, due to some cause inside, is not washing properly. Either your wash rod is wrong or there is an obstruction in the pipe valve; it is not properly opened. Perhaps the strainer system has become clogged and does not properly distribute the water. The sand may have become so foul that it needs a caustic bath. If you are taking forty-five minutes to an hour to wash sand, something should be done to repair the filter plant.

*Mr. Norman*—The filter has only been reconstructed within the last three or four months. New sand and new valves have been put in. It ought to be in good condition.

*Mr. Weedon*—I would like to go into that with you at the plant and I will find out from you where the difficulty is, for that is altogether too long.

*Mr. Davis*—What do you wash with—filtered water or raw water?

*Mr. Weedon*—That depends on how bad your raw water is usually. If your raw water is extremely bad it wouldn't do to introduce that water into your strainer system and up into the stone, because it would take too long to wash it out in the rewash. I say rewash to distinguish from back-wash. If there is available a big storage for filtered water, it is usually more satisfactory to wash with filtered water, because when you do back-wash there is nothing to wash out or rewash, which is turning water back into the filter and letting it run through the sand and out through the rewash. That rewash is a very important point—the more it is stirred up the better you wash. When you pass the water through that sand, it collects a little dirt—the little dirt that is left in the water. I would prefer to wash with filtered water. If it is not available, use raw water. It takes quite a lot of water to wash a filter, possibly eight minutes with 9 or 10 gallons per square foot per minute. With that amount it assists materially; and, as I mentioned before, we do introduce an air system That is merely to economize on wash water. The other point, of course, is to prevent mixing the gravel and sand.

*Mr. Davis*—I would like to ask another questiion. We

find it very difficult to remove oil from the filter. Are you able to wash oil from the filter?

*Mr. Weedon*—Wool grease and oil are our enemies. I have in mind right now one or two cases—one in particular where we had a lot of trouble with wool grease coming down from the plants. If wool grease gets in in any large quantity it will smooth over the grains of sand and is very difficult to wash off. A chemical treatment is needed to take it out. The most satisfactory way is to put strainers and all other mechanical means in the sediment basin to keep it out, and replace them occasionally. I have a case right now, due to an accident at one of the plants. The fireman went to sleep and left a pump running from a large oil tank. He slept into the night and there was about 3 inches of crude oil over the river the next morning, and this got into one of our filters. If you ever saw a mess in a filter, that was it. We are trying to get it out without replacing everything. The safest way is to keep it out. Washing of the filter can't be done too carefully. The treatment of the sand cannot be given too much consideration. Sometimes it is necessary to give a caustic treatment every few minutes. You should put into the filter about a quarter of a pound of caustic soda per square foot of area, filter into the bed and gradually open your filter—your waste valve—and let that drop down to the level of sand for twelve to twenty-four hours. It is a long job. That means a very thorough back-wash to keep it washed out. That one treatment would clean up almost any sand.

*Mr. Davis*—Does it require a warm caustic?

*Mr. Weedon*—No, only a cold caustic soda. In a very bad job you can put on a steam connection. I wish I had some samples. It is just a difference between a very dirty, slimy sand and white sand. It is not necessary to heat the caustic, but in some cases it might be desirable. I cannot stress too strongly the question of keeping sand clean. The washing controls the rate of filtration; it controls the time between washes; and really the sand in the filter is about the most important thing about the plant. Many plants formerly troubled are not troubled now, due to proper washing facilities. This picture shows one of the oldest style filters; it is a wash-rate filter. The Frisco Company has four or five plants and everyone has that filter. They filter about sixty million gallons.

*Mr. Davis*—You said that practically any workman could handle the plant. With all that information given now, I think it would be quite difficult for the ordinary workman. And also it would make the expense more than $1.

*Mr. Weedon*—That means taking out in a plant like, say, one on the Blackstone River, farther up; I don't mean down here in Pawtucket, where some woolen mills want to take out twenty-five parts per million of turbidity, and where they haven't a word to say about twenty-five or thirty of color. I can describe a job we are just putting in. They have a workman running it now. What we have to do is put in a pressure filter of 25 or 30 gallons a day. Our workman was sent for one day and now all they have to do is close the valve to the mill and open the valve for the wash water and let it wash back through the filter for a number of minutes. We tell them when to shut off the washer, then open the waste valve and let it go four or five minutes, shut the waste valve and open the valve back into the mill. That is the simplest filtration job. Take a plant where we are going to feed in alum and soda ash, where it is going into a sedimentation basin and is going to run by gravitation to the filter bed and the pH is put at 6.6. A plant reports trouble with rust in the pipe and we raise it to 7.2. It doesn't require any chemist; but in some plants the chemist is in charge, and rightly so. He can tell the man in charge that he wants the color held at a certain point this afternoon, and the man can follow instructions. Of course, I should have to insist that you put on a man with reasonable intelligence. The tests for pH are very simple. If you are interested in that, we can do that for you. A pH can be run on a filtered water or raw water in two minutes and the chemical feeds are graduated in eight or ten parts per minute. It is a very simple thing to do and does not require a graduate from a textile school or chemist to run it. We do want a reasonable amount of brains, though, to run that filter plant.

*Mr. Wingate*—You spoke about coagulation on one side. You were not using alum—I thought you said it should be on the alkaline side for alum?

*Mr. Weedon*—I spoke of having an alkalinity of about 8 or 10. You must have it on the acid side of the pH scale. That might start an argument with some chemists. For our alkalinity we would use a Methyl Orange test; we would say Methyl Orange alkali. Well, now, that water on the pH scale is distinctly on the acid side, and water with a pH of 5 is ten times as acid as pH 6. pH 4 is 100 times as acid in intensity as pH 6; but the coagulation for water, the standard coagulant, is alum, and if you feed alum into the water it is simply sulphate of aluminate. That is all you have; it goes through, you have alum in treated water and you have to feed in the like amount of alkaline salt to form aluminate hydrate, and that is what the sand holds up—what we call floc. That takes place generally between the limits of 5.8 and 6.6 or 6.8, and is decidedly on the acid side on this particular scale.

*Mr. Wingate*—With the Methyl Orange test it would come on the alkaline test?

*Mr. Weedon*—Yes, certainly. That could cause quite a lot of discussion. There is one man here who uses it, so far as I can determine, more successfully than any other plant in existence. He has worked out his own

plant in conjunction with that of the manufactured sodium aluminate. I happen to know that that man, who had used other chemicals—standard chemicals—in his sedimentation basin before he went there, has cut his chemical bill from $15 to $20 a day by working out a formula with sodium aluminate and ash instead of alum—or whatever it was he was using. He deserves a lot of credit for having worked that out.

*Mr. Wingate*—Do you think you can remove color as well with sodium aluminate

*Mr. Weedon*—If my chemist made a report to you that with alum and soda ash, feed 2 grains of alum and 1½ grains of ash, it would simply be a question of juggling the alkilinity content. A pH of 6.4 based on alum and ash, to be fed on either the acid side or the other side, probably with a sodium aluminate feed, you would raise it possibly two or three or four tenths and it would require a little less correction in your filter water. In one particular case I have in mind the color is apparently satisfactory and the man in charge has no difficulty whatever in holding his color down to 5. He would look askance at a report that came in as high as 10. I think 5 is considered perfect. I think, too, that there is a possibility of a slight increase of hardness with the use of sodium aluminate—a very slight one, I mean, like one or two parts per million. There are certain waters that sodium aluminate won't work at all. That is a problem which has to be worked out in each individual water. The control of your water at the right pH for proper coagulation must be determined in the laboratory. If your raw water happens to be on the alkaline side, due to some sort of dumping on the river above you, do not try to bring it down with sodium aluminate. It is cheaper to feed sulphuric acid. Then get your coagulation after you get your pH. Alum drops the pH slightly anyway. It increases acidity.

*Mr. Wingate*—In coagulating—feeding in with the pump—will the substance be out in time?

*Mr. Weedon*—There are many jobs where we can feed alum or alum and ash. I don't know of any jobs feeding sodium aluminate into a pressure line. After this test, the water comes out crystal clear. There are waters in which we must have a sedimentation period ranging from one hour up. There are a number of waters filtered perfectly satisfactorily every day by a pressure feed. You can use ammonia and alum in lump form, which is expensive.

*Mr. Wingate*—Can you use sulphate?

*Mr. Weedon*—If you use a cheap alum it is not satisfactory. You must get sodium alum. This here [indicating] is the river valve. If the depth is about one-half a grain and on this side, the outgoing side is where you would control it. A half grain seems to be fine enough. Usually your alum feed would be 1 to 1½ grains. Probably it might be three, but not very often. Besides this, you might have to put in soda with the alum. If the pH is too low, your pH corrector, which is a certain kind of soda, is put in on the outgoing side of the filter and you feed in a little sal soda. It is very accurate and in any waters will give you absolutely crystal clear water. In any negotiation for a filter I would simply explain what you would have to pay for, more or less, on your first cost. If your raw water had a color of 75 or 80 and had to be brought down to 20, that pressure pump would answer in most cases. But it wouldn't be satisfactory if you had to get rid of $CO_2$.

*Mr. Searle*—I would like to know what kind of apparatus you consider best to determine pH from.

*Mr. Weedon*—I think the La Motte people put out the simplest apparatus. La Motte has a set of pH blocks ranging from 2 up to 11 or 12, with color ranging to take approximately 1½ points around where we would ordinarily work, right in here [indicating]; a purple just below and a red just above; so that for any ordinary range of work where you wanted just one set—that is, a water filtration set—a primo blue set would be used. A Bromcresol Purple ranges from 5.2 and a Bromthymol Blue goes from 6 to 7.6. We have here—and I think you will be interested to see it—a La Motte set, and I am going to ask Mr. Howe, from the Glenlyon Print Works, to come up here and demonstrate it to show how simple it is. He will show us what the pH of water is and what it looks like.

. . . [A demonstration by Mr. Howe.]

*Mr. Searle*—What I am mostly interested in is a color comparison. A layman who never had any training in color would not be able to determine that, do you think? One would need to have a good eye for color.

*Mr. Weedon*—It isn't so hard, in daylight particularly. The colors are sufficiently different so that you can tell. I am not a chemist; I am a mechanical engineer; and I would not hesitate to go on a job with this set right here and tell a man what the pH of his water is, and I don't claim to be a chemist.

*Mr Searle*—Dr. Strachan is not very well taken to La Motte apparatus—he prefers mechanical electrical resistance.

*Mr. Weedon*—I have never seen any other method. That doesn't mean, of course, that there aren't any other methods; but I don't know where they are. [Holding up one of the test tubes.] You can see that comparatively there is a jump of two-tenths in color each time.

*Mr. Howe*—Our water runs about 6 9, and if it keeps in that range it is all right. If the pH is at 6.8 we add 6 grains of alum and put in enough soda to get 6.4. We try to keep between 6.2 and 6.8 or 6.9, so it really doesn't make much difference to Bernie if we have 6.2 or 6.5. We have a plant equal to three million gallons a day, and

I should say that our chemist must go up only every other day for half an hour in the morning, simply to supervise. This chap who is running our plant never saw a filter plant until two years ago. He is, of course, one above the ordinary training.

*Mr. Weedon*—The determination of those colors is very necessary to the result you want to get. There are plants running—and running successfully—which work without a pH test. We don't like to have them do it, but it is done. The result you want to get is, the condition of the water when passing it through sand will leave in the sand the coagulating color. It must get into that condition, and the best way we know of being sure is this perfectly simple test, and one doesn't need a trained eye to determine it. If any of you desire you can come up and try it yourself. There is another test—the residual aluminate test. That test can be run and guaranteed to indicate one-tenth of a part in a million. In fact, a man, after a little practice, would have to be rather careless not to show one-tenth part of a million of alum if that much is present. We would be glad to demonstrate that test if you wish.

## Hydrogen Ion Control by the Colorimetric Method*

By F. R. McCrumb
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Practically anyone who has worked in a mill or laboratory has at some time or other made use of indicators to estimate the acidity or alkalinity of a solution. Tests with litmus, Congo Red, turmeric and phenolphthalein, although qualitative only and frequently extremely inaccurate, are in reality rough determinations of the hydrogen ion concentration.

The use of litmus paper has perhaps been the most common method of testing solutions for acidity or alkalinity. This paper contains a dyestuff, Azolitmin, which is red in acid and blue in alkaline solutions. In making tests with litmus paper, if the paper is changed to a deep red the solution is said to be strongly acid; if the paper is faintly red the solution is called weakly acid. In a similar manner alkaline solutions are designated as strongly or weakly alkaline, depending on the depth of blue color shown by the litmus paper.

The same method is sometimes followed with Congo Red paper, except that here the colors are reversed, since the dyestuff in this case is blue in acid and red in alkaline solutions.

*A paper delivered before the January meeting of the New York Section of the American Association of Textile Chemists and Colorists, held at the Machinery Club, New York, January 27.

It is apparent that such tests will be subject to considerable error, in that different observers will have different ideas as to the intensity of the color. This single factor prevents any reliable control by this method.

### The Hydrogen Ion Scale

To secure accurate control of the acidity or alkalinity by the colorimetric method, the scheme must be placed upon a quantitative basis. This is what has been done, and, instead of vague and indefinite terms, like "slightly

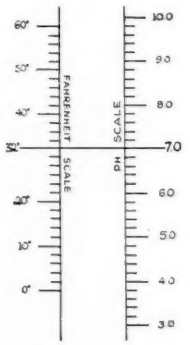



Fig. 1

acid" or "strongly alkaline," definite values are substituted which can be recorded and duplicated at any time by the same or different workers. In other words, use is made of a quantitative scale of acidity and alkalinity.

Probably the scale most familiar to us all is that of temperature, as shown by the Fahrenheit thermometer. As an illustration, let us assume that values above and below 32°, the freezing point of water, represent varying degrees of heat and coldness, respectively. Values above 32°, such as 35°, 40°, 50°, etc., denote increasing heat as these values *increase*. Values below 32°, such as 25°, 20°, 10°, etc., denote increasing coldness as the values *decrease*.

Similarly the degree of acidity or alkalinity of a solution is expressed by a scale known as the hydrogen ion scale. However, the units on this scale are called pH values instead of "degrees Fahrenheit." On the hydrogen ion, or pH, scale a value of 7.0 represents exact neutrality; that is, a solution with a pH of 7.0 is neither acid nor alkaline.

Following the illustration of the thermometer given above, any values higher than pH 7.0, such as 7.2, 7.4, 8.0, 9.0, 10.0, etc., denote alkalinity, the degree of alkalinity *increasing* as the numbers *increase*. Any values lower than pH 7.0, such as 6.8, 6.6, 6.0, 4.0, 2.0, etc., denote acidity, the degree of acidity *increasing* as the numbers *decrease*. Thus solutions of acids tend to have low

pH values, and solutions of alkalies tend to have high pH values.

Suppose, for example, we have a solution at pH 8.8. If acid be added the pH will fall to 8.6, 8.0, 7.0, 6.0, 5.0, etc., and the decrease will depend upon the amount of acid added. If alkali be added the pH will rise to 9.0, 9.2, 10.0, 11.0, 12.0, etc., and the increase will depend upon the amount of alkali added.

For practical purposes a pH value can simply be regarded as a number denoting the degree of acidity or alkalinity of a solution and an operator will soon learn to use this term just as he does the term "degree Fahrenheit." It is apparent that one can make accurate temperature readings without knowing the derivation of the term "degree Fahrenheit." Similarly a knowledge of the meaning of the term pH is not essential in order to use the method for measuring acidity and alkalinity.

## The Indicators

The determination of the hydrogen ion concentration, or pH, by the colorimetric method is based on the fact that certain indicator dyes give different colors in solutions of different acidity or alkalinity. Since the various dyes undergo change at different points on the pH scale, it is possible by the use of a series of such dyes to make pH measurements at any point on the scale.

There are a large number of indicator dyes that undergo change when the acidity or alkalinity is varied. Clark in his book on "The Determination of Hydrogen Ions" lists 158 compounds that have been recommended as indicators. It should be noted here that many of these are not at all satisfactory for practical use. It is only by careful examination that reliable indicators can be chosen for this purpose.

In the following table is given a complete set of indicators to cover the entire pH range from 0.2 to 13.6. For each indicator is given its pH range and color change. These are shown even more clearly by means of the chart on page 153.

TABLE I

| Indicator | pH Range | Color Change |
|---|---|---|
| Acid Cresol Red | 0.2— 1.8 | Red—Yellow |
| Meta Cresol Purple | 1.2— 2.8 | Red—Yellow |
| La Motte Yellow | 2.6— 4.2 | Red—Yellow |
| Bromphenol Blue | 3.0— 4.6 | Yellow—Blue |
| Bromcresol Green | 4.0— 5.6 | Yellow—Blue |
| Chlorphenol Red | 5.2— 6.8 | Yellow—Red |
| Bromthymol Blue | 6.0— 7.6 | Yellow—Blue |
| Phenol Red | 6.8— 8.4 | Yellow—Red |
| Cresol Red | 7.2— 8.8 | Yellow—Red |
| Thymol Blue | 8.0— 9.6 | Yellow—Blue |
| La Motte Purple | 9.6—11.2 | Purple—Red |
| La Motte Nitro Yellow | 10.0—11.6 | Yellow—Orange |
| La Motte Sulfo Orange | 11.0—12.6 | Yellow—Deep Orange |
| La Motte Violet | 12.0—13.6 | Red—Blue |

Litmus gives a red color in an acid solution (pH 4.6 or under) and a blue color in an alkaline solution (pH 8.4 or over). In a similar manner Bromthymol Blue gives a yellow color in a solution having a pH of 6.0 or under and a blue color in a solution having a pH of 7.6 or over.

The point has often been brought up as to why some of the common indicators, such as litmus and phenolphthalein, are not used. This query is best answered by enumerating the qualifications of a satisfactory hydrogen ion indicator. Such an indicator must be available in a pure form; it must be stable in solution; it must be sensitive to hydrogen or hydroxyl ions only; it must give sharp and well-defined color changes, and this necessitates covering a relatively short pH range; and, finally, it must not be subject to errors due to variations in temperature.

Litmus covers a range of 3.8 pH units, from 4.6 to 8.4, as compared with 1.6 units, the range of the indicators listed above. An actual comparison of litmus with Bromthymol Blue at pH 6.0 and pH 7.0 shows the striking superiority of the latter as a pH indicator. It will be seen that with litmus it is difficult to tell the difference between the colors produced by solutions differing by as much as 1.0 pH unit, whereas with Bromthymol Blue variations as low as 0.1 pH can frequently be determined.

As an illustration of the gross inaccuracies frequently made in testing with litmus paper, some results of actual tests are given in the following table:

TABLE II

| pH | Reaction to Litmus | pH | Reaction to Litmus | pH | Reaction to Litmus |
|---|---|---|---|---|---|
| 6.2 | Acid | 7.0 | Alkaline | 7.4 | Neutral |
| 6.6 | Neutral | 7.0 | Alkaline | 7.4 | Alkaline |
| 6.7 | Neutral | 7.1 | Alkaline | 7.5 | Alkaline |
| 6.8 | Alkaline | 7.1 | Alkaline | 7.8 | Alkaline |
| 6.9 | Alkaline | 7.3 | Alkaline | 7.9 | Neutral |

These results show that pH values of 6.6, 6.7, 7.4 and 7.9 were reported as neutral, whereas 6.8, 6.9 and 7.0 were considered alkaline.

While the common indicators are not considered as satisfactory for pH determinations, they have their uses, of course, in titrations and qualitative tests. For this reason it is interesting to know just where they fall on the pH scale. The following table shows this:

TABLE III

| Indicator | pH Range | Color Change |
|---|---|---|
| Erythrosin | 0.0— 3.6 | Orange—Pink |
| Methyl Orange | 2.9— 4.4 | Red—Yellow |
| Congo Red | 3.0— 5.0 | Blue—Red |
| Methyl Red | 4.4— 6.0 | Red—Yellow |
| Litmus | 4.6— 8.4 | Red—Blue |
| Phenolphthalein | 8.3—10.0 | Colorless—Red |
| Turmeric | 8.0—10.2 | Yellow—Orange |

### Preliminary Measurements

The preliminary tests required before exact determinations can be made will depend on one's knowledge of the constituents present in the solution or material being tested. If, for example, one is degumming silk or scouring wool, naturally the preliminary tests will be made in the region of alkalinity—say, from pH 8.0 to 12.0

If one is using a strong acid in a dye bath he will know that the pH should fall in the acid range—say, from pH 1.5 to pH 3.0, etc.

If we assume that nothing is known about the solution, preliminary measurements can be carried out after the following manner:

Fill a graduated test tube to the mark (10 c.c.) and add 0.5 c.c. of the Bromthymol Blue solution (pH 6.0 to 7.6) by means of the graduated dropper. Bromthymol Blue is used first because it covers the neutral point, having a pH range from 6.0 to 7.6. Thus on the first preliminary test one can tell whether the solution is acid, neutral or alkaline. A yellow color will indicate that the pH is 6.0 or under; a blue color that it is 7.6 or over; but a color intermediate, such as yellow-green, green or green-blue, will indicate that the pH is between 6.0 and 7.6. An exact determination can now be made.

If, however, the color is yellow or blue, additional preliminary tests must be made. If yellow, to another 10 c.c. portion add 0.5 c.c. of Bromcresol Green (pH 4.0–5.6). If the resulting color is yellow, the pH is 4.0 or under; if blue, the pH is 5.6 or over; if between yellow and blue, the pH lies between 4.0 and 5.6, and an exact determination can now be made.

In case Bromcresol Green gives a yellow color, another test should be made, using Bromphenol Blue, which is yellow at pH 3.0 and blue at pH 4 6.

If the Bromthymol Blue test shows yellow and the Bromcresol Green test shows blue, it is evident that the pH lies between 5.6 and 6.0, and an exact determination can be made using Chlorphenol Red, which covers the range pH 5.2 to 6.8.

If in the first preliminary test with Bromthymol Blue the color is blue, a test is made with Thymol Blue. This indicator is yellow at pH 8.0 and blue at pH 9.6. If the test portion is blue, the pH is 9.6 or over; if yellow, the pH is 8.0 or under; if between yellow and blue, the pH is between 8 0 and 9.6, and an exact test can be made.

If Bromthymol Blue gives a blue color and Thymol Blue gives a yellow color, the pH probably falls between 7.6 and 8.0, and a determination can be made using Phenol Red, which covers the range pH 6.8 to 8.4.

In making preliminary tests some prefer to make all the tests at one time, and then make the comparisons. This may save some time in case the pH proves elusive; but, of course, if the tests showed a pH between 6.0 and 7.6, all the additional tests would be superfluous.

These preliminary tests may appear boresome, but, in actual practice, once a process has been standardized such tests are rarely necessary. Suppose a certain process has been found to function best between 8.4 and 9.0. The operation is started at, say, 9.0, and in the course of the process the pH decreases; that is, the acidity increases. If the operator knows that he cannot get satisfactory results below 8.4, he takes care that the pH never drops below that value. If it is inadvisable to go above 9.0 he takes care not to exceed this value.

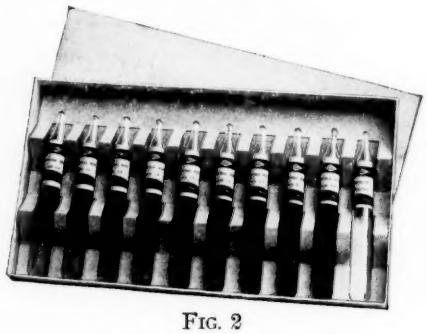


Fig. 2
*La Motte Color Standards*

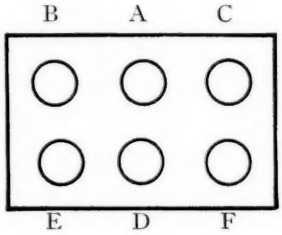
### Exact Measurements

In making exact measurements of the hydrogen ion concentration in industrial processes, we use color standards and comparators.

There is a set of color standards for each indicator in Table I. A set consists of nine standards to cover the pH range of each indicator, or 1.6 pH units in intervals of 0.2 pH. For example, a set of Bromthymol Blue contains the following color standards: 6 0, 6.2, 6.4, 6.6, 6.8, 7.0, 7.2, 7.4 and 7.6. In addition, each set is provided with a tube of distilled water.

The colorimetric method is one of color comparison, and this is best done by means of apparatus especially designed for this purpose. The Block Comparator and Roulette Comparator are such pieces of equipment. The Block Comparator is designed especially as a portable outfit, and hence can be used either in the laboratory or in the plant. A single Block Comparator consists of a set of color standards for any one indicator, such as Bromthymol Blue; four graduated test tubes (10 c.c.) of uniform bore and thickness, and a 50 c.c. bottle of the corresponding indicator. The bottle is fitted with a 0.5 c.c. pipette and nipple for measuring the indicator solution, the nipple serving as a stopper for the bottle. All this equipment is contained in a wooden case, about 8 by 2 by 5¾ inches. When the top of the case is placed over the tubes and fastened down, the set can easily be carried about.

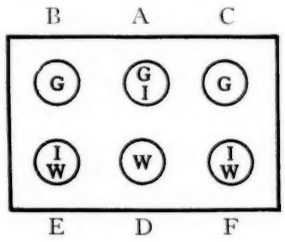
In making measurements the top is removed and used as a comparator. This top contains three slots, covered on one side by a piece of etched glass in a brass holder. The six holes back of the slots in the side of this block will be designated as B, A, C and E, D, F, respectively, as shown in the figure:




Fill three of the graduated test tubes to the mark (10 c.c.) with the liquid to be tested and place them in the holes marked B, A and C. To the middle tube (A) add 0.5 c.c. of Bromthymol Blue Indicator solution from the bottle, by means of the graduated pipette and nipple, and shake the contents thoroughly. Place the tube of distilled water in the hole marked D, and two of the color standards, differing by only 0.2 pH—for example, 6.8 and 7.0—in the two holes E and F. Look through the three pairs of tubes, holding them toward a window or any other source of daylight, and change the color standards if necessary, until the central pair of tubes exactly matches one of the other pairs, or until the color through the central pair lies between the colors of the pairs on either side. Always make sure that the standards placed on either side of the ampoule of distilled water differ by only 0.2 pH; that is, 6.8 and 7.0, 7.0 and 7.2, etc. If an exact match is obtained, the pH of the solution is read off directly from the standard with which the match is obtained. If, however, the color of the central pair of tubes lies between the colors of the pairs on either side, the pH value is taken as the average of the two. For example, if it lies between 6.8 and 7.0 the value is taken as 6.9. The piece of etched glass which is placed over the slots on one side of the block is a great aid in making accurate measurements, as it eliminates the reflection of outside objects in the tubes. The block should be held so that the etched glass is on the side facing the source of light.

FIG. 3
*La Motte Block Comparator*

The reason for the special arrangement of tubes is to eliminate the effects of color and turbidity. Suppose that we are testing a solution that is colored. Let us designate this solution as G, water as W and the indicator as I. Then, in carrying out a test by the method just outlined, the various tubes will be indicated as follows:




From this arrangement it can be seen that any pair of tubes will contain the same color constituents; namely, G, I, W.

Obviously a solution already colored cannot be compared directly with a color standard, but such comparison must be made by observing the color standard through a layer of the colored solution. The tube of distilled water is added in order that the thickness of liquid will be the same in all cases.

Examination of the table of indicators will show that the ranges of many of them overlap. This makes it possible to measure the pH with more than one indicator and thus secure check results. Care should be taken, however, in interpreting tests falling at the extremes of the pH range of any indicator. It should be remembered that values under or over the range may be read incorrectly. Values under 6.0 will be read as 6.0 with Bromthymol Blue.

The Roulette Comparator does not differ greatly from the Block Comparator, the principle followed in making determinations being the same in both cases. However, the Roulette enables one to cover a greater range, make more determinations in a given time, and it also provides a constant source of diffused daylight, so that tests can be made during the day or night under any lighting conditions, just so a source of electric current is available. Since the color standards need never be taken from the drum, the possibility of breakage and the possibility of error, due to inadvertently reading the wrong standard, are reduced to a minimum.

In a metal band, which is mounted on a wooden base, revolves a wooden drum on ball bearings. On the front of the metal band is a slotted block containing three holes In front of the slots in this block is fastened a piece of

etched glass in a brass holder, and in back of the slots is a piece of daylight glass. In the center of the drum is a 40-watt frosted Mazda globe with suitable electrical connections. Around the outer edge of the revolving drum holes are bored. Color standards, alternating with tubes of distilled water, are placed in these holes. The Roulette will hold any three sets of color standards, such as those for Chlorphenol Red (5.2–6.8), Bromthymol Blue (6.0–7.6) and Phenol Red (6.8–8.4).

The method of carrying out the tests is essentially the same with the Roulette Comparator as with the Block. Instead of changing the color standards, the drum is revolved until a match is obtained or the color of the test portion falls between two consecutive standards.

## Meaning of pH

While a knowledge of the meaning of pH is not necessarily essential in the application, technical men prefer to know how these values are derived.

The theory of ionization holds that certain types of chemical compounds are split apart, or dissociated, in aqueous solutions yielding electrically charged particles. These particles, which are known as ions, are responsible for many of the properties of aqueous solutions. When acids are dissociated they produce hydrogen ions, as

$$HA \longleftrightarrow \underline{H^+} + A^-$$

and the acidic nature of an acid solution is determined by the extent to which such dissociation occurs.

Similarly bases produce hydroxyl ions

$$BOH \longleftrightarrow B^+ + \underline{\bar{O}H}$$

and the basic nature of an alkaline solution is determined by the extent to which such dissociation occurs.

Fig. 6
*La Motte Roulette Comparator*

In the case of water, which we can designated by the formula HOH ($H_2O$), it is evident that we have both hydrogen and hydroxyl ions

$$HOH \longleftrightarrow H^+ + \bar{O}H$$

and consequently absolutely pure water is neutral, since neither the acidic agent nor the basic agent is in excess. The extent to which this dissociation takes place in water is extremely small, probably only one molecule in about 555,000,000 being split apart.

Such a system is amenable to the law of mass action, and hence

$$\frac{[H^+] \times [\bar{O}H]}{[HOH]} = K$$

or since HOH is so close to unity

$$[H^+] \times [\bar{O}H] = Kw$$

a constant which is known as the dissociation constant. This has been measured by a variety of methods and found to be

$$\frac{1}{100{,}000{,}000{,}000{,}000} = \frac{1}{10^{14}} = 10^{-14}$$

at 22° C. Consequently the concentration of hydrogen and hydroxyl ions will be

$$\sqrt{Kw}, \text{ or } \frac{1}{10{,}000{,}000} = 10^{-7}$$

The use of such values is so impractical that it was suggested by Sorensen that the concentration be expressed by the logarithm of the reciprocal of the hydrogen ion concentration designated as pH or

$$pH = \log \frac{1}{[H^+]}$$

Since the logarithm of the reciprocal of $\frac{1}{10{,}000{,}000}$ or the hydrogen ion concentration of pure water is 7.0, this is taken as the neutral point.

The relation

$$[H^+] \times [\bar{O}H] = Kw$$

will hold in all aqueous solutions, and consequently it can readily be seen that acid solutions will contain minute concentrations of hydroxyl ions and that alkaline solutions will contain minute concentrations of hydrogen ions. For convenience the hydrogen ion is chosen as the unit of measurement, and one speaks of the hydrogen ion concentration

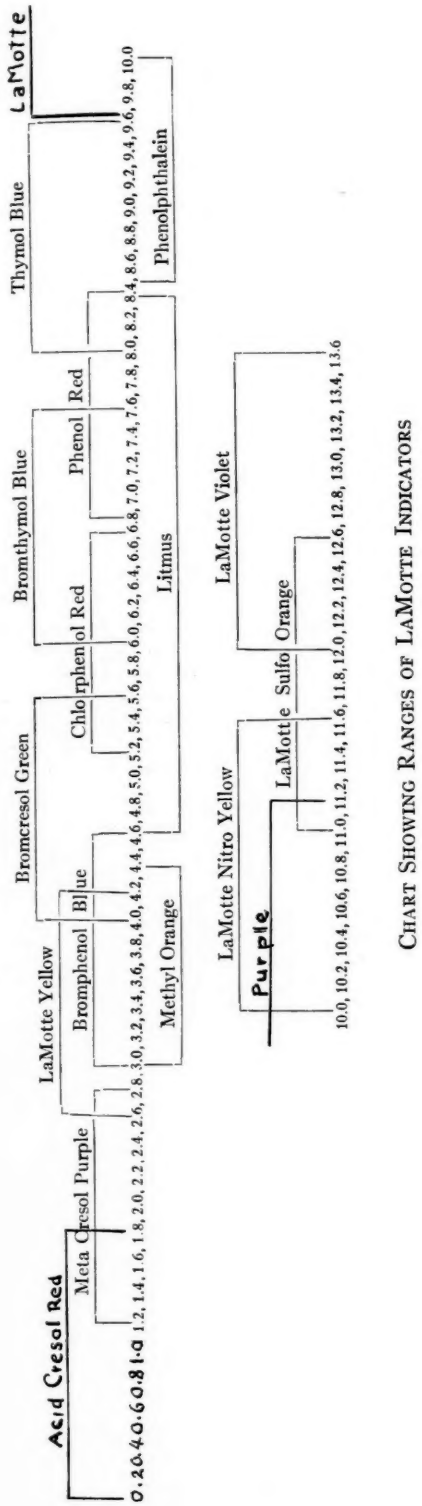

LaMotte
Thymol Blue
Phenolphthalein
Bromthymol Blue
Phenol Red
Bromcresol Green
Chlorphenol Red
Litmus
LaMotte Violet
LaMotte Nitro Yellow
Purple
LaMotte Sulfo Orange
LaMotte Yellow
Bromphenol Blue
Methyl Orange
Meta Cresol Purple
Acid Cresol Red
0.2 0.4 0.6 0.8 1.0 1.2, 1.4, 1.6, 1.8, 2.0, 2.2, 2.4, 2.6, 2.8, 3.0, 3.2, 3.4, 3.6, 3.8, 4.0, 4.2, 4.4, 4.6, 4.8, 5.0, 5.2, 5.4, 5.6, 5.8, 6.0, 6.2, 6.4, 6.6, 6.8, 7.0, 7.2, 7.4, 7.6, 7.8, 8.0, 8.2, 8.4, 8.6, 8.8, 9.0, 9.2, 9.4, 9.6, 9.8, 10.0
10.0, 10.2, 10.4, 10.6, 10.8, 11.0, 11.2, 11.4, 11.6, 11.8, 12.0, 12.2, 12.4, 12.6, 12.8, 13.0, 13.2, 13.4, 13.6

CHART SHOWING RANGES OF LAMOTTE INDICATORS

of a solution of ammonia or caustic soda, even though it is known that the determining agent is really the hydroxyl ion.

One point that should be kept in mind is the degree of acidity corresponding to various pH values. This is shown readily by the following table:

TABLE IV

| pH Values | No. Times $H^+$ (or OH) Exceeds That of Pure Water | |
|---|---|---|
| 0.0 | 10,000,000 | |
| 1.0 | 1,000,000 | |
| 2.0 | 100,000 | |
| 3.0 | 10,000 | H + |
| 4.0 | 1,000 | Acid |
| 5.0 | 100 | |
| 6.0 | 10 | |
| 7.0 | 1 | Pure Water |
| 8.0 | 10 | |
| 9.0 | 100 | — |
| 10.0 | 1,000 | OH |
| 11.0 | 10,000 | Alkaline |
| 12.0 | 100,000 | |
| 13.0 | 1,000,000 | |
| 14.0 | 10,000,000 | |

From the figures it will be readily seen that a solution with a pH of, say, 4.0 will be ten times as acid as one at 5.0, one hundred times as acid as one at pH 6.0, and one thousand times as acid as one at pH 7.0.

If one titrates equal volumes of N/10 solutions of the two acids hydrochloric and acetic, using an N/10 solution of sodium hydroxide with phenolphthalein as indicator, the titers will be the same in each case. This is because titration is a measure of the total acid. It has been shown that in the majority of cases the important factor is not so much the total acid as the actual concentration of hydrogen ions which such acids yield in solution.

This is strikingly illustrated in the case of the two acids just mentioned. In N/10 solution hydrochloric acid is dissociated to the extent of 91.4% at 18° C. This means that out of every 1,000 possible molecules 914 have split apart to form hydrogen and chloride ions, the remaining 86 existing as molecules. If all the hydrochloric acid were dissociated, an N/10 solution would contain 0.1 g. of hydrogen ions per liter. Since only 91.4% is dissociated, such a solution will contain $0.1 \times 0.914 = 0.0914$ g. of hydrogen ions per liter. The normality with respect to hydrogen ions is then

$$\frac{N}{1/0.0914} = \frac{N}{10.94}$$

When an acid solution contains 1 g. of *ionizable* hydrogen per liter, we say it is normal. In a similar way we

can say that a solution which contains 1 g. of *ionized* hydrogen per liter is normal with respect to hydrogen ions. Since $\frac{1}{[H^+]}$ is the reciprocal of the normality of hydrogen ions, in a solution, the pH value can be defined as the logarithm of the denominator expressing the normality of hydrogen ions.

In the case of the N/10 solution of hydrochloric acid just cited, where it has been shown that the solution is N/10.94 with respect to hydrogen ions, the pH is the logarithm of 10.94 or 1.04. If all this acid were dissociated, the normalities with respect to both *ionizable* and *ionized* hydrogen would be identical and the pH would be the logarithm of 10, or 1.00.

An N/10 solution of acetic acid also contains 0.1 g. of *ionizable* hydrogen per liter, but it can be shown by electrical conductivity measurements that at 18° C. only 1.36% is actually ionized. Hence the hydrogen ion concentration is $0.1 \times 0.0136 = 0.00136$ g. per liter. This is equivalent to an N/735 solution of hydrogen ions, and therefore the pH of an N/10 solution of acetic acid is the logarithm of 735, or 2.86.

A comparison of the hydrogen ion concentrations of these two acids shows that N/10 hydrochloric acid contains over sixty-seven times as many hydrogen ions as does N/10 acetic acid.

An illustration sometimes used is that of an army, when the hydrogen can be considered as soldiers. The total acidity shows the total number of men in each army, whereas the hydrogen ion concentration shows only those on the firing line. Where hydrochloric acid has 9,140 out of 10,000 on the firing line, acetic acid has only 136.

Since, at any one temperature, the product of the hydrogen and hydroxyl ions in a solution is constant, it will be seen that if one is known the other can readily be calculated. Sodium hydroxide in N/10 solution is dissociated to the extent of 92.9% at 25° C. The normality with respect to hydroxyl ions is therefore $\frac{N}{1/0.0929}$, or $\frac{N}{10.76}$. Since

$$pH = \log \frac{1}{[H^+]}, \quad pOH = \log \frac{1}{[O\bar{H}]}$$

and for N/10 sodium hydroxide this will be the log of 10.76, or 1.03. The pH of this solution will then be (14.00 — 1.03), or 12.97.

In a solution of N/10 ammonium hydroxide only 1.33% is dissociated at 25° C. The normality with respect to hydroxyl ions is therefore $\frac{N}{1/0.00133}$, or $\frac{N}{752}$, and pOH = log 752, or 2.88. The pH of N/10 ammonium hydroxide at 25° C. is therefore (14.00 — 2.88), or 11.12.

A knowledge of both the reserve and the active acidity or alkalinity is of importance in most processes. Neglect of the latter will frequently lead to difficulty.

Such distinction has long been recognized in a qualitative way, but until the introduction of hydrogen ion concentration methods we were content to account for this by designating some acids and bases as strong and others as weak, even though we knew that the total acidities or alkalinities were identical.

The following table gives some idea of the relationship between total acidity and alkalinity and the pH values. The values are for N/10 solutions, and they are rounded off to the first decimal place:

TABLE V

| | pH |
|---|---|
| Acids: | |
| Hydrochloric acid | 1.0 |
| Sulphuric acid | 1.2 |
| Phosphoric acid | 1.5 |
| Acetic acid | 2.9 |
| Alum | 3.2 |
| Carbonic acid | 3.8 |
| Boric acid | 5.2 |
| Bases: | |
| Sodium bicarbonate | 8.4 |
| Borax | 9.2 |
| Ammonia | 11.1 |
| Sodium carbonate | 11.6 |
| Trisodium phosphate | 12.0 |
| Sodium metasilicate | 12.2 |
| Sodium hydroxide | 13.0 |

A point which often comes up is how can one talk about hydrogen and hydroxyl ions in the case of compounds which contain neither; as, for example, alum [$Al_2(SO_4)_3$], which yields fairly acid solutions, and sodium carbonate [$Na_2CO_3$], which yields fairly basic solutions. The production of hydrogen ions in one case and hydroxyl ions in the other is due to the fact that salts of weak bases and strong acids and of strong bases and weak acids react with the water, and the extent to which this reaction occurs determines the concentration of hydrogen or hydroxyl ions. Such a reaction is termed hydrolysis.

### Action of Buffers or Regulators

If one were dealing with solutions of pure acids and bases it would be possible to calculate the pH in each case if the total concentration and degree of dissociation were known. Rarely, however, do we encounter such solutions in practice. Industrial solutions invariably con-

tain a number of known substances and usually other materials of an unknown nature. Many of these affect the hydrogen ion concentration, acting as buffers or regulators, in that they resist change in pH when acid or alkali is added to solutions containing them.

Acetic acid in aqueous solutions will dissociate slightly, as follows:

$$H\,Ac \longleftrightarrow H^+ + \overline{Ac}$$

but the dissociation of sodium acetate is practically complete, as:

$$NaAc \longleftrightarrow Na^+ + \overline{Ac}$$

This yields a large number of acetate ions, and these tend to tie up the hydrogen ions as undissociated acetic acid. By varying the amount of sodium acetate, one can prepare mixtures having different pH values. Since within practical limits the pH of such a mixture depends largely on the ratio $\frac{HAc}{NaAc}$, dilution within reason has very little effect. This situation is taken advantage of when the pH of very highly colored or turbid solutions must be determined.

The buffering effect of sodium acetate is so pronounced that when acetic acid solutions are titrated with sodium hydroxide the solution becomes alkaline to Methyl Orange long before an equivalent quantity of base is added. The sodium acetate formed soon raises the pH above 4.0, the so-called neutral point of the indicator.

An N/100 solution of sulphuric acid will have a pH of 1.8. The presence of 10% of Glauber salt will increase this to 2.6, showing a lowering of the active acidity, despite the fact that the total acidity has remained unchanged.

In an analogous manner the pH of a solution of caustic soda will be lowered by the presence of borax, as has been pointed out by Dr. Chapin in his work on wool.[2] A solution 0.04 molal with respect to sodium hydroxide had a pH of 12.3 when no borax was present, but borax to the extent of 0.02 molal lowered the pH to 10.6.

If we take 10 c.c. of practically pure water and add merely a drop of an N/10 solution of hydrochloric acid, the hydrogen ion concentration will increase tremendously, the pH decreasing from about 7.0 to 3.0, or less. The same amount of acid added to a standard phosphate buffer mixture of pH 7.0 will cause practically no change.

As long as a solution will transmit light when placed in one of the standard tubes, a colorimetric determination can be made, even if the color or turbidity is fairly high. When light will no longer pass through, the method of dilution must be employed. As has been pointed out previously, most industrial solutions are buffered, and this renders it possible to dilute until a test can be made. Of course, all dilutions must be made with a good grade of distilled water so as to avoid introducing anything which might change the pH.

A practical method of determining the actual effect of dilution is to make a series of dilutions, such as 1:1, 1:2, 1:5, 1:10, etc., and then determine the pH at each dilution. This will show at once whether the method can be applied with safety.

There are very few solutions so highly colored that they cannot be measured by the colorimetric method in a dilution of 1:10, and many industrial solutions will undergo no appreciable change in a dilution of 1:100.

In testing dye baths where the color is too intense, the most practical method is to prepare a buffered dye bath and determine the pH previous to addition of the dyestuff. The exhausted bath can usually be tested without any difficulty.

### Precautionary Measures in Determining pH

It will be readily seen that no unusual precautions need be employed when testing a buffered solution, since it resists change in pH, at least to a reasonable extent. However, with unbuffered solutions considerable care must be taken, especially near the neutral point, since a very small amount of acid or alkali has an enormous influence on the pH.

The first inclination in taking up pH control is to make a determination on the distilled water supply. This determination is not difficult, providing all the factors are taken into consideration. While water of absolute purity will have a pH of 7.0, ordinary distilled water will have a lower pH, due to dissolved carbon dioxide absorbed from the air. Distilled water in equilibrium with the air will ordinarily have a pH of about 5.7. Water from an efficient still, stored in non-soluble glass, will usually have a pH of 6.2–6.4. If such a water is boiled in a pyrex glass flask and then sealed with a soda lime trap to prevent access of carbon dioxide, the pH will be about 6.6–6.8.

In making determinations on unbuffered solutions, the indicator should be mixed with the sample by means of a stirring rod to avoid excessive exposure to the air. The tube should not be shaken. Care should also be taken that the indicator solution itself is neither strongly acid nor alkaline, but as close to neutrality as possible.

While in some cases it is an advantage to be able to use indicator papers, as a rule the use of such papers

is extremely unreliable, particularly when slightly buffered solutions are being tested. With buffered solutions it is possible to secure fairly close approximations, providing satisfactory color standards are available; but the carbon dioxide of the air may introduce an element of error when the solution being tested has little buffer capacity. Test papers are sometimes coated with a layer of material which absorbs only the aqueous portion of a mixture, and this permits tests on pastes and highly turbid mixtures. In using such papers care must be taken that the protective agent itself does not change the reaction. With unbuffered solutions this might result in an appreciable error. If, for example, a test was being made on water, the pH secured might be the pH of the protective coating rather than the pH of the water.

Another method sometimes employed in testing slightly colored or turbid liquids involves the use of capillary test tubes and standards. This method overcomes color and turbidity to a very slight extent, but it is decidedly unreliable in testing solutions only slightly buffered, since it is necessary to mix the indicator with the sample outside of the capillary tube. This gives a maximum of exposure to the carbon dioxide of the air, resulting in considerable inaccuracy when the solution is not buffered.

It should be kept in mind that even if dilution does affect the pH, there are many places where pH control by the colorimetric method can be used if a constant dilution is adhered to. This gives a working figure which in many cases is just as valuable as an accurate pH figure would be.

There are cases in which tests on solid materials have given information of extreme value. An extraction of the material with distilled water is made and the pH of the extract then determined. Such a determination often provides a knowledge of the condition of the material as regards acid or alkali content.

Strong oxidizing agents, such as hypochlorites, will affect the indicators. However, it is possible to make fairly accurate determinations by the colorimetric method if the bleach is first destroyed by the addition of a very small amount of a neutralized solution of hydrogen peroxide. A fairly strong solution of peroxide should be used, since the volume required is then so small that the dilution effect is minimized. Hydrogen peroxide itself does not materially affect the indicators, so that they can be used directly in controlling peroxide bleaching baths.

In the case of a reducing agent such as hydrosulphite, a slow reduction of the indicator occurs at any appreciable concentration, but up to about 0.5% sodium hydrosulphite this decolorization is slow enough to make satisfactory determinations of the pH. If a buffered bath is being used, dilution of a more concentrated hydrosulphite solution with distilled water can be employed.

Since temperature frequently has a marked influence on the degree of ionization, it would be expected that the pH would change with variation in temperature. This actually does occur in many cases, and for this reason pH measurements on a given solution should be made at approximately the same temperature, preferably at room temperature, 68 to 86° F. (20 to 30° C.). In any case, measurements made at one temperature should not be compared with those made at another, unless experiment has shown that the temperature effect for that particular solution is practically negligible. The temperature effect is not due to any inaccuracy in the method, but is an actual change in pH, which will be shown by any method of measurement.

## Applications in the Textile Industry

The amphoteric nature of wool and silk would lead one to believe that hydrogen ion concentration must be one of the important factors in their wet processing. Both have definite isoelectric points in the neighborhood of pH 4.0 to 5.0, where their chemical reactivity is at a minimum.

When silk is initially soaked in an emulsion of oil in water, the stability of the emulsion depends to a large extent on the pH. This is true of emulsions in general.[1]

While the scouring of wool and the degumming of silk require a certain alkalinity, about pH 10.0, to be effective, care must be taken that the pH of the bath does not exceed a safe figure. In both these processes the materials removed from the fibers are excellent buffers or regulators, and this renders it possible to replenish the baths with alkaline reagents, providing the pH is carefully controlled. In the case of wool, if the temperature is kept below 140° F., a pH of 11.0 can be safely maintained.[2—3—4]

In the bleaching of wool, silk and cotton by means of hydrogen peroxide, pH control has been recommended in the literature [5] and is now being used to a considerable extent in practice. A pH in the neighborhood of 10.0 appears to be most satisfactory, although there is still some difference of opinion regarding the exact optimum reaction for the various materials.

Some recent work on the role of pH in the bleaching of cotton with hypochlorite indicates that "within the range of hydrogen ion concentration pH 5.0 to 10.0, characteristic of technical bleach liquors, the maximum rate of oxycellulose formation occurs at the neutral point." [6]

Since the activity of an enzyme depends largely on the hydrogen ion concentration, a desizing process which depends on diastatic action should be particularly amenable to pH control.

In the dyeing of silk and wool it has been demonstrated that the affinity of the fibers for acid dyes increases as the pH decreases, and is probably best at pH 2.0 or under. In the case of basic colors, the affinity increases with increasing pH and the optimum is probably above pH 11.0. However, such an alkalinity obviously could not be employed with silk and wool.[7]

Similarly it has been shown that the affinity of some mordants for acid and basic dyes follows the same rules.[8]

Hydrogen ion control is being applied in a number of fields intimately related to the textile industry. In the purification and softening of water such control has proved to be of considerable value. Coagulation, either with alum or copperas, occurs best at some particular hydrogen ion concentration. In zeolite softening the pH of the brine has an important effect on the efficiency of revivification of the zeolite.

In treating various textile wastes, purification occurs most efficiently at pH 8.0 to 8.2, when copperas is the coagulant, and at 7.2, when alum is used. Vacuum or plate and frame filtration of wool-washing liquors after coagulation failed until proper adjustment of the pH was made, near 7.2, and then was readily carried out.

Hydrogen ion control is being used in laundry work to determine the efficiency of final rinsing, and also to test the extractor water, so that dangerous amounts of sour will not be left in the clothes.

There is need for much additional research to determine the role that hydrogen ion concentration plays in the processes enumerated above, and in others that may be amenable to pH control. There are many other factors to be considered, and hydrogen ion control should not be looked upon as a cure for all the difficulties. It does, however, give promise of becoming a valuable tool in the textile industry.

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### Discussion of the Colorimetric Control of pH Concentration

*W. A. Kingman*—I should like to ask how long those set-up standards in that wooden block, for instance, will keep. Will they fade or will they change color?

*Mr. McCrumb*—Any color standard like that will change with time, but its life depends a whole lot on the treatment it gets and it depends a whole lot on the particular indicator. Lately we have succeeded in improving some of them to a considerable extent, but we guarantee the standards to last at least a year, and in many cases they will last much longer.

*Chairman Hayes*—Are there any further questions?

*Dr. H. C. Chapin*—Mr. McCrumb brought out one point that I think ought to be emphasized, and that is that we don't want to revolutionize any textile processes. We want to measure what you are doing from day to day, and then you will have a record of it. When you get good results you will know why you got them and you will be able to duplicate.

*Chairman Hayes*—Dr. Chapin says that he will be glad to stay here and make a few actual determinations on this instrument for the benefit of anyone who would like to see that.

Are there any further questions?

Unquestionably, we have all enjoyed and benefited by the two papers we have had here to-night. Before we adjourn, I want to personally thank the two speakers, on behalf of the Section, and I shall call for a rising vote of thanks.

. . . A rising vote of thanks was extended to the speakers. (Applause.)

. . . The meeting thereupon adjourned at 10.30 p. m.

## JANUARY MEETING OF THE PIEDMONT SECTION

The January meeting of the Piedmont Section of the American Association of Textile Chemists and Colorists was held at Hylmor Restaurant, in the Jefferson Standard Building at Greensboro, N. C., on Saturday, January 21, 1928.

Papers were presented as follows:

Prof. Albert H. Grimshaw, associate professor of dyeing of North Carolina State College, Raleigh, N. C.: "Methods of Testing Sulphonated Castor Oils for the Determination of Their Fatty Content." A discussion of the paper followed, the leaders in this discussion being Prof. Charles A. Mullin, of Clemson College, S. C.; Mr. Chase, Mr. Southern and Mr. Monarch.

Following this paper and discussion was one presented

by W. R. Smith, of Raleigh, N. C.: "Some Foolproof Dyehouse Methods." The discussion that followed was led by Mr. Lomac, Mr. Nuckolls and Mr. McKenzie.

T. C. King, superintendent of dyeing, bleaching and finishing of Cramerton Mills, Inc., Cramerton, N. C., presented a paper on "Remarks on Rayon Dyeing and Finishing." A discussion of this paper followed, being led by Mr. Dickson and Mr. Sweet.

J. T. Yates, superintendent of the Kerr Bleaching & Finishing Company, Concord, N. C., read a paper on "Bleaching Cotton Piece Goods." The discussion that followed was led by W. H. Woodhead, of the Gregg Dyeing Company, Granitville, S. C., and Julian Robertson, of the North Carolina Finishing Company.

During the dinner a musical program was furnished by Miss Gwendolyn Mitchell, of the Greensboro College for Women, and Fielding Frye. Mrs. Powell accompanied at the piano.

### Business Meeting

A motion was made by Mr. Whitney that a Technical Committee be appointed to answer technical questions for members of this Section. It was suggested that this Committee consist of a bleacher, a chemist, a yarn dyer, a piece dyer and a piece goods finisher. There was also to be appointed an alternate for each of these members of the committee. The motion was seconded by Mr. Hunt, voted on and carried. The motion was amended as follows:

The names of the members of the Committee are to be known only to the Chairman and Secretary of the Section, and no member of the Committee is to have any knowledge of any other member of the Committee except his alternates. This amendment was voted upon and carried.

H. A. Barnes, Chairman, appointed a committee to survey the Piedmont Section for all persons eligible for membership, whether they be eligible for Junior membership, Senior membership or the new Associate membership. J. D. Sandridge, of the E. I. du Pont Company, was made Chairman of the committee, it being his duty to survey the section east of the Yadken River; J. I. White, of the National Aniline Company, to survey the section west of the Yadken River and east of the Broad River; Tom Nuckolls, of the Piedmont Flush Mills, Inc., to survey the section east of Broad River and to the Georgia line. This committee is to report on its survey at the April meeting, which will be held at Greenville, S. C.

W. R. Smith, of Raleigh, N. C., suggested that the members give Mr. Hunt, Mr. Barnes and Mr. Stone a rising vote of thanks for their efforts and hard work, to which may be attributed a share of the Section's success.

There were 113 members present at the dinner and approximately 135 listened to the presentation of the papers and the discussions that followed.

The paper by Professor Grimshaw is published below. The others will appear in a later issue of the Proceedings.

D. S. Moss, *Secretary.*

## Methods of Testing Sulphonated Castor Oils for the Determination of Their Fatty Content

By Albert H. Grimshaw
*Associate Professor of Dyeing, North Carolina State College Textile School*

When Mr. Stone requested Dean Thomas Nelson of North Carolina State College Textile School to name someone to talk on the above subject, the work was turned over to us in the Dyeing and Finishing Department.

You all are familiar with sulphonated oils and know that they come to the plant labeled as 50%, 75%, etc., sulphonated. With this in mind, we wrote to many firms making the products and to plants using the products asking them to furnish data as to the meaning of this method of naming, and also to furnish tests as performed in their own labs.

At this time we would like to thank these firms and plants for their replies; among those who have given us permission to use their names are the following: Jacques Wolf & Co., Kali Manufacturing Company, Bosson & Lane, Kem Products Company, National Oil Products Company, Onyx Oil & Chemical Company, Standard Chemical Products, Inc., A. Klipstein & Co. However, the mill chemists were so modest that I am not at liberty to disclose their names.

### The Meaning of the Percentage

Here are a few of the answers in regard to the naming of the compound:

No. 1: "I believe that you will find among the trade that the term 50% Sulphonated Castor Oil means an oil containing 50% of water and 50% of sulphonated oil, which when you deduct the ash, glycerine, etc., means a total fatty content of about 43%. Similarly, 75% would contain 25% of water, about 65% fatty matter and about 10% non-fatty impurities. You will understand, of course, that these impurities are absolutely necessary in the manufacture of sulphonated oils and cannot be totally removed."

No. 2 writes: "These materials are generally sold on cold test, which would make a 50% oil contain in actual fatty matter approximately 41.4%; the 75%, 60.14%, and the 85%, 67.64%. Therefore, the cold

test method is, as you no doubt surmise, a volumetric test, and not one which shows actual content of fat."

No. 3 writes: "The figures do not indicate or have no relation to the actual total fatty acids contained in the oil. The results above mentioned are obtained by a so-called volumetric test: using sulphuric acid, brine solution and heat in a graduated flask. This test is employed by the manufacturers because of its simplicity and the short time consumed in making the test. This will give a result indicating the actual sulphoricinoleates contained in the oil by volume. However, this figure does not indicate the total fats in the mixture, but it is easy to compute the total fatty acids from the volumetric results by using the factor 0.81. In other words, the result obtained by the volumetric test, or what is sometimes known as the separation test, multiplied by 0.81 will give the actual total fatty acids present."

No. 4 writes: "The oils are sold both ways and we quote prices according to the two different specifications—whichever one is asked for by the buyer. Some customers ask for 75% sulphonated oil and they interpret this to mean 75% of total ricinoleo-sulphonic acid and soda; in other words, 25% of water. Other consumers ask prices based on total fatty acids. In other words, they ask for a price on sulphonated castor oil—50% total fats—and we quote them accordingly. An oil with 50% total fats would have about 58% total oil; in other words, approximately 40% of water."

No. 5 writes: "Fifty per cent usually means an oil of 50% moisture content, the balance consisting of neutral oil, sulphonated oil, fatty acids, combined alkali, combined $SO_3$ and ash."

No. 6 writes: "The real meaning of 50% or 75% fat with regard to a sulphonated oil, in so far as the trade is concerned, is that the oil contains not over 50 or 25% water, as the case may be. The 50 or 75%, therefore, includes the actual fat plus the combined $SO_3$ plus the sodium sulphate that is present from neutralizing the oil."

No. 7 writes: "We have always based our strengths of sulphonated castor oil upon the amount of oil used in the product, not on the fatty acid content. Inasmuch as castor oil contains glycerine, which is lost in the process, the fatty acid test is, of course, lower than the quoted strength. Our method, however, has been followed by most manufacturers and seems to be the practical way, as the cost is based on the amount of oil used."

## What Authorities Say

We will now quote a few remarks from various authorities. The first is from Molinari: "Ricinoleic acid, $CH_3.(CH_2)5.CH(OH)2.CH_2.CH:CH.(CH_2)7.CO_2H$, constitutes, in the form of glycerine, the greater proportion of castor oil. By treating castor oil slowly with cold concentrated sulphuric acid, 'ricinosulphuric acid' is obtained. By treating with concentrated sulphuric acid, Grun obtained ricinolein—sulphonic acid: $C_{17}H_{32}(O.SO_3H).CO_2H$; by hot water this acid is hydrolyzed with the separation of sulphuric acid and formation of a condensed ester, 'ricinolein-ricinoleic ester,' $C_{17}H_{32}(OH).CO.O.C_{17}H_{32}.CO_2H$, the condensation taking place between the carboxyl group of one molecule and the hydroxyl of another.

"Ricinosulphuric acid or its sodium or ammonium salt is of importance in Turkey Red oil, since when the latter is diluted with water it serves to keep in a state of solution the castor oil or other unaltered oil always found in greater or larger quantity in Turkey Red oil. The complete analysis of a good sulphoricinate gave 58% of total fat, composed of 47% of insoluble fatty acids, 1.5% of neutral fats and 9.5% of fatty sulpho-acids soluble in water."

Lewkowitsch writes as follows: "Sulphonated oil can be resolved into two portions by dissolving it in ether, washing with brine until it is free from sulphuric acid, and then shaking out repeatedly with water. On treating the united aqueous solutions with sodium chloride the water-*soluble* portion separates as an oily layer. On evaporating off the ether from the ethereal solution the water-*insoluble* portion is obtained. Benedikt and Ulzer stated that the *water-soluble* portion, in the case of Turkey Red oil prepared from castor oil, consists of *ricinoleo-sulphuric* acid, which is miscible with water in all proportions. From aqueous solutions the ricinoleo-sulphuric acid is precipitated as a heavy oily layer by brine, moderately dilute sulphuric acid, or hydrochloric acid. If, however, the free acid be boiled with dilute hydrochloric or sulphuric acid, part of it is decomposed into ricinoleic and sulphuric acids, while another part is converted into ricinoleic anhydride."

Grun and Wetterkamp state: "On boiling ricinoleo-sulphuric acid with water in the presence of a mineral acid, the decomposition is considerably accelerated and the formation of esters proceeds beyond the formation of an inner anhydride to probably form a lactide of the formula: $C_{17}H_{32}.O.CO.CO.O.C_{17}.H_{32}$.

"The water-*insoluble* portion of castor Turkey Red oil consists chiefly of free ricinoleic acid and small quantities of neutral 'unacted on' oil, and also of anhydrides of ricinoleic acid."

Scheurer-Kestner say that castor Turkey Red oil consists of ricinoleo-sulphuric acid and of mono-ricinoleic acid and di-ricinoleic acids.

Juillard claims polymerization proceeds further to form di-, tri-, tetra- and penta-ricinoleic acids. He also says that Turkey Red oil is a mixture of varying proportions

of poly-ricinoleic acids, of alkali salts of mono- and poly-ricinoleo-sulphuric acids, of anhydrides of the latter acids, and of their products of decomposition.

Bogajewaky says that the commercial product contains (a) in the water-*soluble* portion, ammonia or soda salts of the acids ($HO.SO_2.OC_{17}H_{32}.CO_3$), ($HO.C_{17}H_{32}.CO_2).C_3H_5.(OH)$, and $HO.C_{17}H_{32}.CO_2H$; (b) in the ether-soluble portion, unchanged castor oil and salts of mixed castor oil fatty acids.

Turkey Red oil is valued on the amount of *total fatty matter* in the sample. This comprises the water-*insoluble* portion obtained on adding a mineral acid to the oil, whereby the fatty acids, hydroxy acids and neutral oils are separated, as also the hydroxylated acids obtained on decomposing the soluble sulphuric esters of the fatty acids.

For analysis Lewkowitsch recommends the methods of Benedikt and the one of Finsler modified by Breinl, both of which will be described in a moment.

Knecht, in his "Manual of Dyeing," states: "Turkey Red oil is not a true sulphonic acid; it belongs, according to its whole behavior, to the class of sulphuric ethers. Ricinoleic acid differs essentially from oleic acid, inasmuch as it contains a hydroxyl group in addition to the carboxyl group. When sulphuric acid acts on tri-ricinolein 'castor oil' or on ricinoleic acid, it does not form a sulphate by addition, as is believed to be the case with triolein, but acts on the hydroxyl group; *a true etherification takes place with the separation of water, and a non-saturated compound is formed.*

"The presence of glycerin in the commercial oil is incontestable, but it is unnecessary to make it enter into the composition of the body, as it is easily eliminated without any modification of the properties of the oil."

Andes says: "Glycerin is not completely separated in the making of Turkey Red oil, one moiety remaining in the oil and increasing its solubility and capacity of emulsification."

## Methods of Testing Compared

Now that we have spoken of the various theories, let us pass on to the actual testing. In reply to our inquiries as to methods used in the plants and mills, we received about twenty-five methods. We have tried out fifteen or so of these and will now try to show you how they are performed and why results varied.

Dr. Browne, of our department, has had charge of these tests and has been assisted by Messrs. Field, Gryder and Howard, who are students in the Textile School. Dr. Browne ran all of these tests and the students checked him with parallel tests. Where results did not check, a third run was made.

The tests were made on three oils from three different makers. Our results will show comparative values with the various tests.

With the first group of tests we will list the methods in which a separation takes place by use of mineral acids, "A" being volumetric and "B" being gravimetric. Under the second group will be listed methods of extraction using ether.

Lewkowitsch says of the boiling with mineral acids: "The total fatty matter forms the water-insoluble portion consisting of fatty acids, hydroxy acids and neutral oil, as also the hydroxylated acids obtained on decomposing the soluble sulphuric acid esters of the fatty acids. In the extraction method the total fatty matter thus formed is less than the sulphonated oil dissolved in the water, by the amount of sulphuric acid split off on boiling with hydrochloric acid." One firm uses both methods and writes as follows: "Volumetric or separation results multiplied by f 0.81 will give the total fatty acids present, as in this test sulpho-compounds are present. In the extraction test the results give actual fatty acids." Grun and others do not agree as to the compounds obtained by action of mineral acids. With the above in mind, it seems best to use the results of these tests in a comparative manner, and not to show actual percentages of any one specific organic compound.

## Tests or Methods

No. 1. *Cold Test Method.*—Eighty cubic centimeters of sulphuric acid, 20° Be., in a graduated bottle or cylinder; add 20 c.c. of the oil to be tested; replace the stopper and shake contents well. Then let stand until all the sulphated oil has risen to the surface and become quite clear. The volume of the liberated sulphated oil is then read off. The number of cubic centimeters multiplied by five gives the percentage of the sulphated oil.

No. 7.—Ten cubic centimeters of the oil are taken and washed into a long-necked flask of about 150 c.c. capacity, the neck of the flask being graduated up to 10 c.c. Add to the flask about 10 c.c. of a saturated salt solution, then add 5 c.c. of concentrated sulphuric acid, agitating the mixture thoroughly. Finally add sufficient concentrated salt solution to bring the fatty layer within the graduations on the neck of the flask. Place the flask in a water bath and heat at a boiling temperature until fatty layer becomes clear or transparent. Reduce to room temperature and read off the volume of the separated oil and calculate to a percentage basis. This gives a result indicating actual sulpho-ricinoleates contained in the oil by volume. However, this does not indicate the total fats in the mixture, but it is easy to compute the total fatty acids from the volumetric results by using the factor 0.81. In other words, the results obtained by the volumetric test, or what is sometimes known as the separation test, multiplied by 0.81 will give actual fatty acids present.

No. 13. *Rough Method.*—Definite amount of sample is placed in a graduated cylinder or test tube, and to this is added about twice as much sulphuric acid of about 20% strength. This is shaken up thoroughly for a minute or two, and the tube or cylinder is then placed in a large beaker of water, which is kept at a boil for about an hour. At the end of this time practically all the fat will have risen to the top of the tube, but still contains a considerable amount of mechanically trapped moisture. However, if the tube is left without being disturbed until the following day a very close approximation of the fat content will be obtained by reading the volume of the oil separated and dividing by the original amount of oil taken. If the tube is graduated in fractions of an inch, and 2 inches of oil were originally taken, from which, let us say, 1⅜ inches of oil will separate, the fat content of the oil would be between 68 and 69%. The important features of this method are keeping the separated oil near the boiling point for a considerable time and taking the reading the following day. If an ordinary volume separation is made cold and read off within a few minutes, a much higher percentage of fat would be shown than is actually present. This is especially true of oils and fats of a high melting point. By the hot method the underlying acid solution at the time of reading the fat volume should be perfectly clear and care should be taken to see that no globules of fat remain adhering to the glass. The above method is, of course, not accurate except within about 2%.

No. 15. *Quick Method.*—Thirty grams of sample rinsed with about 70 c.c. of hot water into a flask of about 200 c.c. capacity with a neck graduated into one-fifth or one-tenth c.c. Add 25 c.c. of sulphuric acid of a specific gravity of 1.563, or 52° Be.; heat to boiling, with frequent shaking until the fatty matter forms a clear transparent layer. A hot concentrated solution of common salt or Glauber salt is next added carefully to bring the oily layer into the neck of the flask. After standing half an hour the volume of the fat is read off. The number of cubic centimeters multiplied by 3.33 corresponds to the total fatty matter expressed as per cent. A correction should, however, be made on account of the specific gravity of the fat being below one; usually 0.945.

No. 16. *Simple Comparative Test.*—Take two graduated wells measuring in 1 or 2 c.c. to 250 c.c. Into each pour 50 c.c. from each sample and add 50 c.c. water. Then add 10 c.c. muriatic acid and stir thoroughly with glass rods. Place the wells in a warm place on radiator or register and let remain for five to six hours. Measure the oil that comes to the top. Get percentage.

No. 27 (another quick method but not very accurate).—Take a glass-stoppered cylinder of about 250 c.c. and graduated to read 0.5 c.c. or finer, and add 100 c.c. oil and 10 to 20 c.c. concentrated hydrochloric acid; shake well and then add saturated sodium chloride solution till the liquid comes to within a few graduations of the top. Allow to stand a few hours and read off the fatty layer. This reading represents the amount of fat, by volume, in the sample.

*Results of Volumetric Tests*

| Test | Sample 1 | Sample 2 | Sample 3 |
|---|---|---|---|
| 7 | 43.5% | 67.3% | 43.5% |
| 15 | 43.4 | 67.6 | 47.3 |
| 13 | 53.4 | 76.7 | 51.7 |
| 16 | 50.0 | 80.0 | 60.0 |
| 1 | 52.0 | 89.0 | 59.0 |
| 27 | 55.0 | 85.0 | 65.0 |
| Average | 49.5 | 75.3 | 54.4 |

### Tests with a Cream Separator

F. E. Busby has suggested using a Babcock cream separator; so we tried out two of the volumetric methods, using a Babcock. The handle was turned at 70 r.p.m. This not only did the work quicker but also gave much clearer solutions than in the methods where the solutions were allowed to stand overnight. The globules of oil were not noticeable on the sides; also only one layer of oil was present. With the other methods, especially with No. 2 sample, there was a middle emulsified layer. Using the Babcock with methods Nos. 1 and 27, without allowing the solutions to stand, we obtained the following results:

| Test | Sample 1 | Sample 2 | Sample 3 |
|---|---|---|---|
| B27 | 49.0% | 76.0% | 63.0% |
| B1 | 53.5 | 75.0 | 57.5 |
| Average | 51.25 | 75.5 | 60.25 |

### Gravimetric Tests

No. 2. *Japan Wax Test.*—Weigh 50 c.c. beaker and small glass stirring rod. Weigh in 10 grams of sample. Add hot water to dissolve the oil. Place the beaker on the water bath, add 4 c.c. of concentrated hydrochloric acid and continue heating until fatty acids are free on the surface. Now add 6 grams of Japan wax and continue heating until the wax is melted. Cool and remove cake, formed of wax and oil, by using the glass rod. Bottom of cake must be dried as much as possible by use of filter paper. Dry and weigh. Weight of cake minus weight of wax equals weight of oil. Multiply by 10 and get percentage, as 10 grams of sample were used.

No. 5. *Webster.*—Five grams of sample plus 5 grams of beeswax and 100 c.c. of hot water; add excess of concentrated hydrochloric acid and salt solution. Heat until all beeswax is dissolved, and let cool. Remove beeswax and fat cake, rinse in cold water, and dry in desicator overnight and weigh. Usual method to get percentage.

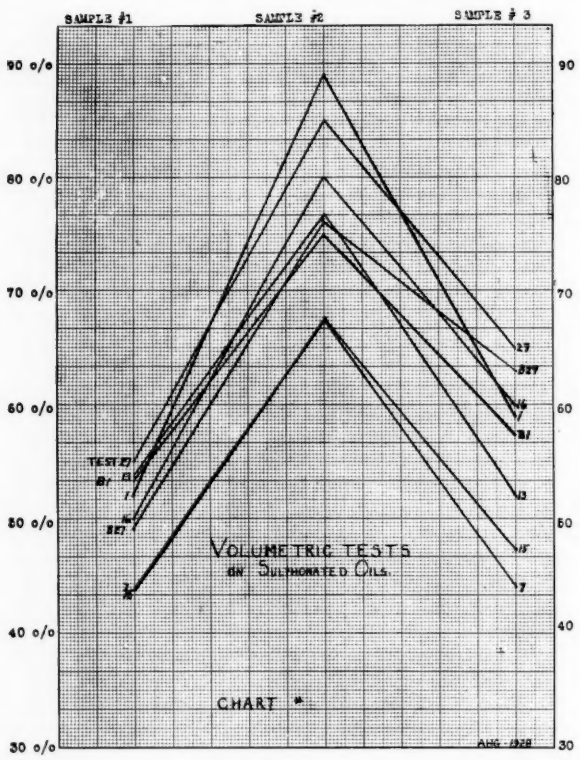



CHART No. 1

No. 6. *Am. P.*—Fifty grams sample diluted to 1,000 c.c.; add sulphuric acid and 20 grams of stearic acid. Boil and cool, let stand overnight, and weigh. [Note: We only weigh to hundredths.] Compute in regular manner. Fairly accurate and serves our purpose of giving comparative results.

No. 9. *K.*—Ten grams of sample in small beaker and dissolve in 25 to 40 c.c. distilled water. Add 10 c.c. CP hydrochloric acid and heat for fifteen to twenty minutes, or until all fats come to the top. Cool in the ice box. If fats will not solidify, add 10 grams of paraffin to the hot solution of the melted fats in the acid bath, and when separated let stand in the ice box as before. Weigh and compute. [Note: At school this method was unsuccessful, as the oil did not melt and stay in the paraffin.]

No. 19. *Benedikt.*—Four grams of sample in 20 c.c. hot water. Make slightly alkaline with ammonia, and then add 15 c.c. of 50 per cent sulphuric acid. Ten grams of beeswax or stearic acid, *not paraffin,* are then added and the mixture heated until a clear, fatty layer has separated on top. After cooling, the hard cake is separated carefully, remelted in hot water, cooled, separated, and dried and weighed, and percentage computed.

*Results of Gravimetric Tests*

| Test | Sample 1 | Sample 2 | Sample 3 |
|---|---|---|---|
| 5—Beeswax ..... | 38.6% | 61.9% | 43.5% |
| 2—Japan wax ..... | 39.1 | 60.6 | 42.7 |
| 6—Stearic ..... | 40.0 | 60.2 | 43.9 |
| 19—Beeswax ..... | 42.5 | 71.7 | 46.5 |
| Average ..... | 40.1 | 61.1 | 44.1 |

Now we come to the extraction tests in which ether is used.

No. 10.—Ten grams sample in small beaker; add about 100 c.c. of water and about 10 c.c. of concentrated hydrochloric acid; heat on steam bath for about an hour. Transfer the contents of the beaker to a separatory funnel, washing the beaker thoroughly with hot distilled water. Cool contents of the funnel and extract with about 75 c.c. of ether. Allow to stand until a clear separation occurs. Draw off the bottom layer of acid water and discard. Wash the ether extract with several portions of cold distilled water and transfer the ether extract to a weighed flask. Connect the flask to a condenser and distill off the ether, using a water bath. Dry the residue in an oven at 100° C. to constant weight and weigh as fatty acids. The weights of the fats multiplied by 100

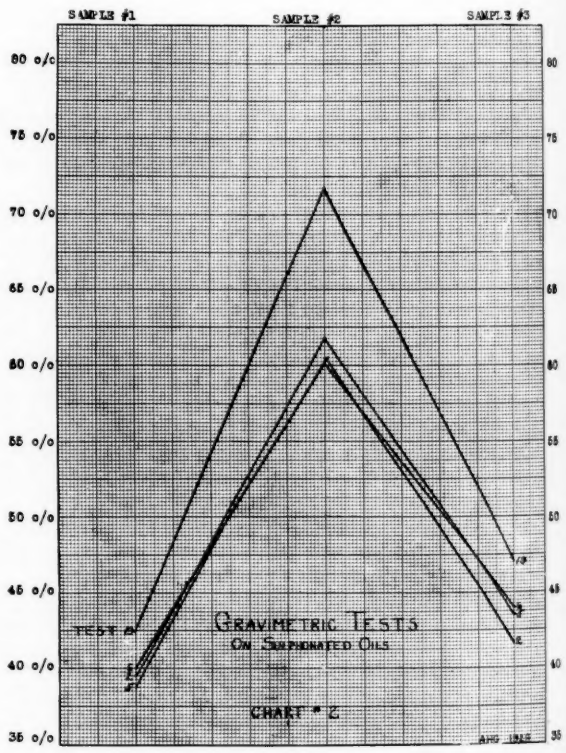



CHART No. 2

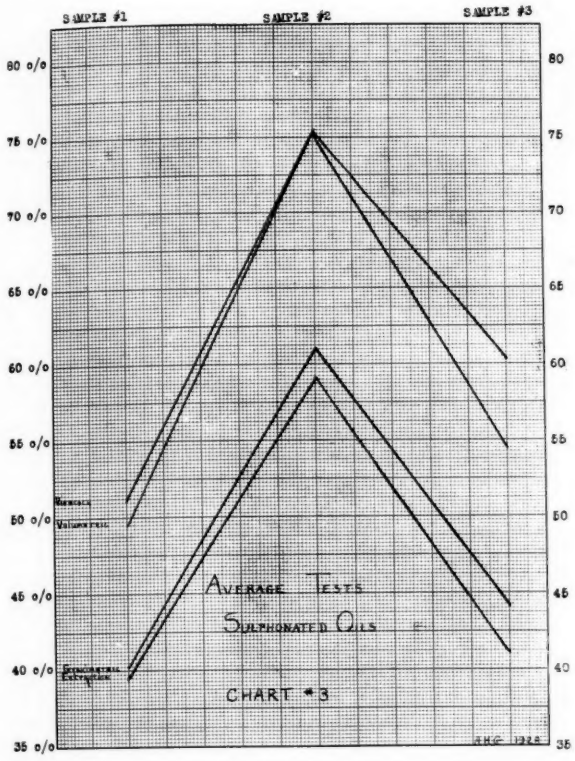



CHART No. 3

and divided by the weight of the sample will give percentage of fatty acids.

No. 11.—Five to ten grams dissolved in warm water. Add 5 c.c. hydrochloric acid and boil for half an hour. Cool and pour into a separatory funnel and extract with ether; common salt or Glauber salt may be used to hasten the separation. Allow to stand until separated. Draw off the acid solution. Run ether extract into a tared flask, distill off ether, and weigh residual oil and compute percentage.

No. 17. *US.*—Weigh 4 grams into Erlenmeyer and boil forty minutes with 30 c.c. hydrochloric acid 1 to 15, using glass beads to prevent bumping. Cool, transfer to separatory funnel and extract the fat with ether. Wash ethereal layer not less than three times with 75 c.c. water, using a little hydrochloric acid if necessary to break emulsion. Transfer to a tared beaker, evaporate, dry, cool and weigh. Then calculate percentage.

*Results of Extraction Tests*

| Test | Sample 1 | Sample 2 | Sample 3 |
|---|---|---|---|
| 10 | 38.0% | 57.7% | 39.6% |
| 11 | 39.2 | 59.8 | 41.4 |
| 17 | 41.5 | 59.9 | 42.6 |
| Average | 39.6 | 59.1 | 41.2 |

Four charts have been prepared from these tables. On chart No. 1 we have plotted the curves of the volumetric tests, including the ones with the Babcock machine. Tests 7 and 15 show the lowest results, whereas 27 and 1 have the highest results. The Babcock tests were run with these two, and that may account somewhat for the high resulting average in the Babcock tests.

Chart No. 2 contains curves of gravimetric tests. Note that Nos. 2, 5 and 6 are closely bunched, whereas No. 19, for some reason, is much higher.

Chart No. 4 shows curves of extraction tests. These are also closely bunched.

Chart No. 3 shows the curves of the average of the four ways of testing. There is a wide difference between the two volumetric results and the gravimetric result, whereas the gravimetric and the extraction curves run very close together. Now, one author states that multiplying by the factor 0.81 will give actual fatty acids when applied to volumetric tests, and such results will be nearly made in doing such figuring with the volumetric results here and then comparing with the extraction figures.

## Summary

The mills that sent in replies did not seem to care so much about the actual fatty acids as they did about the

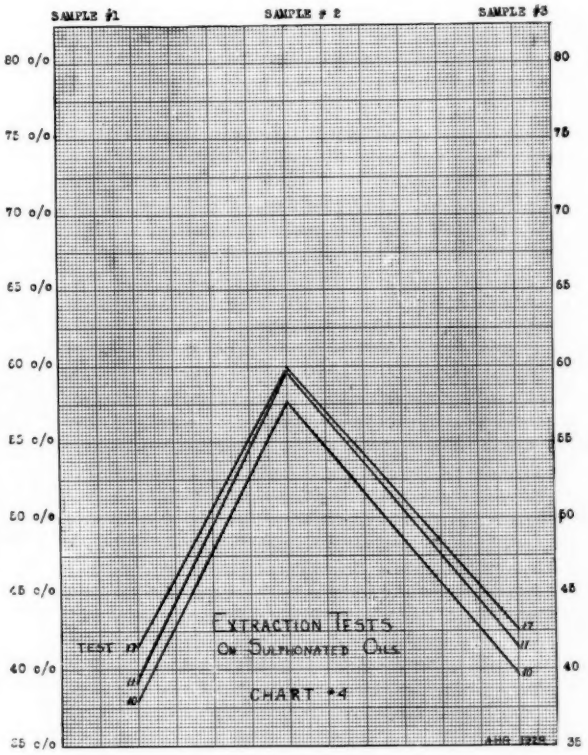


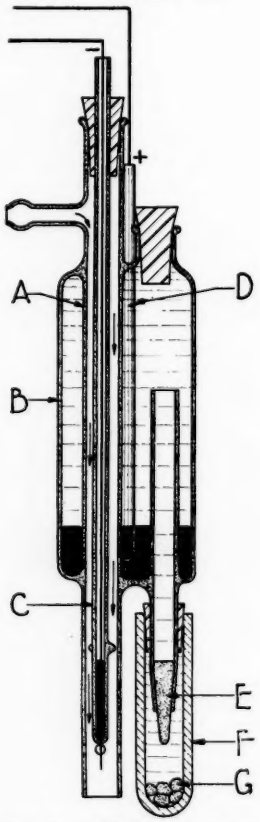

CHART No. 4

comparative strength when compared with one of their own standards; for that reason they used the volumetric tests. However, the manufacturers used both the gravimetric and the extraction tests.

The ease with which results were obtained with the Babcock machine seems to justify using it if only comparative results are desired. As to which volumetric test to use, that would depend upon the apparatus on hand; but from tests which we have shown it should be an easy matter for the mill man to make these tests. The manufacturers, of course, will make their tests in a more scientific manner.

## Errata

In Dr. Chapin's paper entitled "Electrical Methods of Measuring and Automatically Controlling Chemicals" as published in the Proceedings of the February 20 issue of the REPORTER, in the last paragraph on page 123, specific references were made to an illustration which was inadvertently omitted. The following illustration and paragraph should be substituted for the last paragraph on page 123:




*Cell for pH Measurement*

Mercury, calomel and saturated potassium chloride solution go into the glass compartment (B). From the bottom of this protrudes a tube with ground glass stopper (E) just tight enough to hold solution but pass electric current. Over this tube fits a porous earthenware thimble (F) full of saturated potassium chloride solution. Through a long tube (A) penetrating the cell goes a glass holder (C) for one of the various wire electrodes, and through this same tube may be passed hydrogen, entering at side arm near the top. The electrode holder is a fine glass tube with electrode sealed through its tip. A little mercury inside the holder makes contact between the electrode and a fine copper wire thrust in from the top. This wire goes to the potentiometer, as does also a wire (D) similarly connected with the mercury of the calomel compartment. The electric circuit is completed by dipping the projecting wire electrode and earthenware thimble into the solution under examination.

## Applicants for Membership

*Active Membership*

Farlow, W. A., color matcher and dyer, Deep River Mills, Randleman, N. C. Sponsors: D. S. Moss and W. M. Hunt.

Forstall, Walton C., chemist, S. Sanford & Sons, Inc., Amsterdam, N. Y. Sponsors: G. R. Pensel and W. E. Hadley.

Freedman, Ephraim, chemist, Standards Bureau of R. H. Macy & Co., New York, N. Y. Sponsors: H. F. Herrmann and D. P. Knowland.

Goldsmith, Jr., William, foreman of dyeing, Lowell Bleachery No. 2, Griffin, Ga. Sponsors: C. A. Jones and Tom Taylor.

Mease, Arch J., salesman and demonstrator, Newport Chemical Works, Inc., Oak Park, Ill. Sponsors: R. P. Cole and Edward Weber.

Murray, Clyde, dyer and bleacher, Mayfair Mills, Burlington, N. C. Sponsors: D. S. Moss and W. M. Hunt.

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Pardue, Wm. A., Jr., student, North Carolina State College Textile School, Raleigh, N. C. Sponsors: A. H. Grimshaw and J. D. Sandridge.

# AMERICAN DYESTUFF REPORTER

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## NO LONGER VISIONARY

A LOOK into the world of the future often amazes us by its novelty, as much as a backward glance into the past surprises us by what it reveals of the quaint world of our ancestors. We may find prophecies as hard to swallow as some of the tales with which history is adorned. Would the dyer of twenty years ago, we wonder, have believed the picture of the Modern Dyehouse which is drawn for us in the article published elsewhere in this issue? Rather, he would most likely have regarded it as not less visionary than the fantasies of Jules Verne. The division of the day's work that he customarily handled unaided, among a large staff of trained specialists; the schematic arrangement of the plant machinery and processes; the standardizing of shades and materials —all this would be as inconceivable to the dyer of a generation ago as the flight of Lindbergh.

But "the world do move," for better or for worse, depending on the particular school of philosophy that views the changes. Transport your dyer of a quarter century ago to the floor of a modern dyehouse and he would believe that he was floating through the ecstacies of an opium dream. His first reaction, perhaps, would be a yearning for the good old days when a man learned his trade without a thought for the new-fangle theories; when he had all his work not only under his eye but under his own hands, and knew his shades and drugs like the alphabet. The speed and apparent confusion of the new system would bewilder him for a time. But to bring the gleam of appreciation into his eye we would have merely to explain to him the basic principle on which this elaborate systematizing and standardizing operates. The new-day dyehouse would then look to him more like a skillfully designed machine, well oiled, silent running, and efficient down to the smallest bolt and screw.

It is not to be assumed, however, that the ideal has become a firmly established reality: that the majority of dyehouses in the country now operating are "modern" in the sense implied by the author of our article. But scientific management is no longer an idler's vision, nor efficiency the butt of the jokesmith. The larger, more progressive mills have set the mark by their adoption of the most approved methods of dyehouse operation, by their installation of the most modern equipment and their application of the newest theories of control to their processes. Their followers will, in a few short years, become legion.

## "SILK ONLY PART—"

IT takes but very little tin to make all the difference between approval and censure of the practice of silk weighting. If we may paraphrase a famous advertising slogan, a few per cent make "a whale of a difference" to those who oppose overweighting.

The "whale" in this case, however, has become a "wail" within recent months, and it has been in the ranks of the silk dyers that the wailing has sounded loudest. And not without reason; for the dyers who apply the weighting process, and who have long studied its effects on the fabric, feel it is their province to warn against excesses.

Their protests have been made, from at least one quarter, to the cutting-up trade. Interest in the subject of weighting extends to many groups outside of that trade, however. When the effects of excessive weight rebound, they hit all branches of the silk industry, from the fabric producer and the dyer to the retail merchant; and the mutter of complaint disturbs even the air beyond, for the dry-cleaners have also come out vigorously against it; and they, in turn, are in intimate contact with the consuming public.

Those who indiscreetly have pushed the practice of weighting across the line of safety and common sense probably did not anticipate the fuss they have stirred up. It is now too late to silence the wail, for it has become more serious than merely an irritating noise. In some sections of the trade, as we intimated, the complaints against overweighting have prompted certain groups of technical men to study the actual chemical effects of the process on the wearing properties of the fabric, to correlate the available data on the process and to establish from this certain interesting conclusions as to the relation of the loading to dye fading and other signs of deterioration.

The dyers and cleaners deserve some of the credit for establishing these facts; the research staff of their trade association, working in the laboratories of the Government Bureau of Standards, issued more than a year ago an authoritative report on "The Effects of Dry-Cleaning on Silks," in which the action of the weighting process was discussed at some length.

Supported in their contentions by such sound technical study of excessive weighting, some of the silk dyers have not hesitated to be candid in their assertions against those who demand still heavier fabrics. A group of Paterson dyers, meeting recently at the offices of the Silk Associa-

tion of America, agreed to maintain a sensible, moderate standard of weighting and to curb to the best of their ability the insistent demand for more "stiffness" in silk fabrics.

But few have been as admirably outspoken on the subject as Albert Blum, of the United Piece Dye Works, who has consistently opposed the growing tendency toward excessively loaded fabrics and has urged manufacturers, retailers and others to join in advocating a moderate percentage. In a characteristic letter published a short time ago he said: "I decry this demand for hand and stiffness which will make goods crease and break when made up into dresses and will eventually drive women away from silk fabrics."

That thought is particularly timely in view of the greater popularity of rayon goods to-day. Here is a source of competition that cannot be brushed lightly aside. The thumbs of the buying public may be turned down for silk by the extent to which weighting is applied beyond what Mr. Blum terms the "danger point."

We would almost be willing to wager, however, that all the thought and study and co-operation which have gone into the campaign against heavier silks will not equal in effect the blasting wit that is released in the words of this parody which, adding a melodious note to the wail, "made the rafters ring" at the recent annual dinner of the Silk Association:

You made me what I am to-day, I hope you're satisfied;
You've weighed me up and up until my weave is sorely tried.
Remember that you promised to "go light" when it began;
But you've broken my heart, I'm silk only part, And mostly a tin can.

## PHILADELPHIA LAUNDRIES TO EDUCATE CONSUMERS

### Laundry Service, Research and Textile Information Will Be Featured in Talks

False notions concerning the work laundries are doing will be dispelled in the minds of consumers if the plan of educational work laid down by the Philadelphia Laundryowners' Exchange is carried out carefully and systematically.

What is termed the first effort of its kind in this country will be directed by Mrs. Edith W. Pierce, who has recently been appointed by the Laundryowners' Exchange. Popular talks on the service rendered to the community by the modern laundry will be given before various civic, church and social organizations and to domestic science teachers and classes. What has resulted in modernizing the laundry through the efforts of the Laundryowners' National Association and its fellowship at the Mellon Institute of Industrial Research will be pointed out to these groups of consumers and others who come in contact directly or indirectly with laundries.

The educational talks that are being planned will include full discussion of the research and other work undertaken by the laundry industry for the purpose of attaining a higher degree of professional knowledge of the maintenance of textile products. The scientific study of processing methods, of colors and of fabrics will be outlined, in order that the housewife may understand the problems of the laundry as related to the selection, care and washing of the textiles she buys.

The prime object of this educational campaign, it is declared in the announcement, is to correct the many prevalent mistaken ideas regarding the service of laundries and to present the actual facts to the consumer. It is further pointed out that this should also be of benefit to the producer and retailer of textiles in the light of the co-operative efforts between the laundryowners, manufacturers and retail stores at the recent conferences held in Washington and Cincinnati.

## ANDRAE RETURNS TO HOECHST

Friends of A. R. Andrae, New York, sales manager for General Dyestuff Corporation, will be interested to know that he is giving up this position and will sail March 14 on the steamship Albert Ballin to take charge of the American Department at the Hoechst plant of the I. G.

Mr. Andrae became associated with H. A. Metz & Co., in 1906. From 1907 until 1909 he was Chicago manager, and from 1910 to 1912 Philadelphia manager for this concern. From 1912 until the formation of the General Dyestuff Corporation he acted as New York sales manager for H. A. Metz & Co. His many friends throughout dyestuff circles will wish him well in his new field.

## TECHNICAL LIBRARY FOR SALE

Prof. Charles S. Doggett, for many years head of the Department of Textile Chemistry and Dyeing at Clemson College, S. C., now retired, is anxious to dispose of his rather extensive technical library, which he has accumulated over a long period of years. In addition to many standard and some unusual volumes on the subjects of textile chemistry and dyeing, Professor Doggett also has files of trade periodicals, including the AMERICAN DYESTUFF REPORTER, and a large number of sample books and technical treatises issued by various foreign and domestic dyestuff manufacturing concerns. Professor Doggett will be glad to consider the sale of the library as a whole or any special volumes which may be desired. Correspondence should be addressed to him at Clemson College, S. C.

## EXTRACTORIALS

I cannot conceive, in this age of scientific attainment, with the fruits of research so evident in other industries, why research in the fields of textiles has not been heavily endowed and vigorously pushed.—*M. D. C. Crawford.*

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When an industry establishes standards of size, dimension, grade or quality for the products of its members, and not only maintains those standards but certifies to the public its products conform to those standards, that industry is practising self-government and much credit is due that industry for its endeavor.—*Technical Bulletin, Bureau of Standards.*

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A laundryowner's life is made up of doing a multitude of little things, over and over again; he passes through his plant thousands of articles—there is much detail—much care must be taken to keep things straight so that all his customers may have the correct count and all their things back. One little thing done wrong or gone astray makes more stir and loss than a thousand which have taken the right course.—*George Peck, in the Starchroom Laundry Journal.*

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Ideals wear thin with years, and ogres, when lived with, as Beauty discovered, are apt to lose their ogrishness. The practice of one generation seems sometimes to be obtained by reversing the panaceas of the previous generation. Twenty-five years ago a threatened industrial combine was sufficient to arouse fury, while now an industry without its combines has become a subject for reproach.—*From "A Business Man's Diary," Manchester Guardian Commercial.*

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We feel reasonably sure that American ingenuity, American science and American capital can hold its own in world markets, if an effective tariff be used to protect the great American consuming market against unfair foreign competition, if the industry co-operates with the universities in furthering research and training men, and if the American chemical industry will wholeheartedly cooperate, one unit with another, in entrenching itself and perfecting its program of development.—*Editorial, Industrial and Engineering Chemistry.*

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It is possible to get the results from combining the duties of dyer and finisher that are obtainable through division. Either position in the average plant calls for the best knowledge and skill and the undivided attention of the man in charge. Much, of course, depends on the size and character of the work carried on in the two departments.—*Textile Review.*

Business carried to a high degree of efficiency may be either an art or a science; it all depends on the business and the degree of efficiency.—*Charles Carpenter, in The Houghton Line.*

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It is quite possible as investigation and research are pursued that new types of fibers will be produced which are a combination of one of the older vegetable or animal fibers with artificial silk. There is a statement in the Textile Zeitung of Berlin that a German company has produced a yarn of this class. It is made of very fine cotton with a coating of artificial silk.—*British Silk Journal.*

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Spectroscopy is at once one of the simplest and one of the most complicated subjects within the realm of natural science. While the full theoretical explanation involves the latest theories of atomic structure, the quantum theory, and other abstruse principles, its application to practical problems is, with modern equipment, convenient and simple, and it metes out its benefits with great generosity.—*S. Judd Lewis, D.Sc., in the Journal of the Society of Dyers and Colourists.*

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Education is not learning things by rote—it is not remembering a lot of rules like most young fellows think it is. Education is simply understanding. When you understand anything you have learned it for all time to come.—*Old-Timer, in Cotton.*

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There has been more money spent by the chemical yarn manufacturers of the United States in the past four years in research, so as to bring about higher perfection in their products, than by any other group of textile manufacturers here or abroad.—*Editorial in Rayon.*

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## MARKET SURVEY ADVISED BY BROAD SILK COMMITTEE

Every silk manufacturer would be working with a better knowledge of market conditions if he made a careful study of percentages of stocks in relation to profitable sales, according to a report submitted by its executive committee to the Broad Silk Manufacturers' Division of the Silk Association of America. If only as a precaution in preparing for the remainder of the present season, such study would be justified in view of the conditions that tend to overstocking which have existed in the industry in previous years.

An intelligent survey of market and inventory conditions in the current season would be a pertinent one for the silk manufacturer, in the light of past experience, declared Paul C. Derby, chairman of the Broad Silk Division, at its recent meeting. Mr. Derby pointed out that loss of profits through overstocking would be less of a problem to the industry if individual manufacturers applied themselves to a study of the statistics affecting their sales.

## THE MODERN DYEHOUSE

(*Continued from page* 140)

peratures. Moisture content of the pastes is periodically standardized by the chemist. Rigid control of the drugroom removes one variable from dyehouse operation.

A very convenient system of weights, promoting both speed and accuracy, is the decimal pound, which adapts the easy mental calculation of the metric system to the customary plant use of the English system. Calculations of costs, percentages, adds, etc., are simplified, often requiring only the moving of a decimal point. Matchers find it easy to estimate adds in percentages of the starting formula. All weighed materials are double checked before issuance to the operatives, and repeat formulas are checked against the original to prevent expensive errors in production. Delivery of material is made only upon written requisition. When possible, materials are issued in the proper condition for immediate use. The quantities of materials per shade are accurately recorded with impartiality. The psychological effect of this impartial check acts to keep all formulas to minimum quantities. Preliminary cost data, tabulated by the drugroom as it accumulates, is forwarded to the cost department daily. A properly operated drugroom prevents deterioration of raw materials, renders possible accurate material costs, prevents many production mishaps, and increases the productive time of men and machines.

### Reducing Matching to a Science

Routine shade matching is delegated to moderately paid men with the ability to match shades quickly and accurately. They are freed from all the other details of dyehouse operation, and the increased opportunity to concentrate on one task produces better matches in less time. In case the matcher doubts a specific match the trial patch is submitted to the dyehouse superintendent for advice. All shades are passed upon after the first drying by the dyehouse superintendent; release of the lot for further processing requiring his approval. If any change in shade is anticipated during specific finishing operations, the superintendent's approval precedes further processing of the lot. By this progressive approval of questionable shades, unnecessary processing of imperfect goods is eliminated. Preliminary formulas on new shades, on new fabrics, or by new dyeing processes, are furnished to the matcher by the superintendent of dyeing so that production time is not unnecessarily wasted. All new shades are matched in the laboratory, and the formula thus furnished, while it may not hold exactly true in the plant, will indicate the quantities needed and will designate the exact dyes required, so that production time will be saved and possible seconds prevented.

A file of master formulas is kept in the matcher's office, and a duplicate file is in the drugroom. These formulas have been standardized by repetition in production. Swatches of goods in all stages of processing, from dyeing to the finished product, attached to the formula cards, provide accurate data for the matcher. Tentative formula cards contain all available data, and when repetition proves the formula to be correct, master formula cards are issued.

The primary standard shades are mounted in light proof folders and protected from atmospheric deterioration. Standards which cannot easily be replaced, which are unusually fugitive, or which may be expected to receive very frequent use, are matched by the laboratory and these secondary standards used for routine matching. Periodic checking of the secondary standards against the primary standards permits replacement upon evidence of perceptible change.

The matcher's office receives abundant north light, free from reflected components coming from adjacent structures. The interior is dead black to minimize reflected light. Daylight and ordinary artificial light lamps are enclosed in dead black booths.

To secure maximum machine and labor production is the task of floor men supervising the mechanical details of production. As specified in writing by the matcher, they supply the machines with cloth, checking lot number, yardage, and the superficial condition of the cloth. If the machine speed is variable, they adjust it to correspond with the matcher's instructions. They remove the completed work from the machines and convey it to the next department. For padder work they run the trial patches from the auxiliary tub with the aid of a miniature padder, thus minimizing the non-productive time of the large padder. They complete the jig patches for the jig operators and submit them to the matcher. Their function is to relieve the shade matcher of much detail, and the productive labor of all except productive tasks. Production efficiency of men and machines is consequently high.

### The Human Element

The phase of dyehouse operation which has received the least attention up to the present time is the human element. From a merely callous business viewpoint it pays to study the human side of plant operation. Many industries have taken a broader viewpoint and have felt a certain responsibility towards their employees which is not solely due to a stark desire for direct profit.

A properly installed, well operated incentive system, preferably task and bonus, achieves a many-sided goal. The employees acquire a real ability to work, habits of industry are inculcated, their self-respect rises, their co-operation becomes whole hearted, and they become more valuable assets to the plant

and to the community. The plant secures a fully adequate direct financial profit due to the lower labor cost per unit. The men receive higher pay than they otherwise could merit.

A recapitulation of the salient points in the modern dyehouse shows:

1. Only one high-priced executive; a man of training and ability commensurate with the remuneration.
2. Specialization of operations into functions supervised by moderately paid experts.
3. High grade, contented employees.
4. Accurate determination of costs.
5. Technical superiority resulting in:
   - High quality product.
   - Minimum seconds.
   - Minimum material waste.
   - Satisfied customers.
6. Maximum production at minimum cost.

## RECENT LITERATURE

### Identification and Analysis of Dyes

*Allen's Commercial Organic Analysis: Vol. VI—Colorimetry, Dyes and Coloring Matters, Synthetic Dyestuffs and Analysis of Coloring Matters.* Edited by Samuel S. Sadtler, Elbert C. Lathrop and C. Ainsworth Mitchell. Fifth Edition, Revised and Partly Rewritten. 658 pages; illustrated. P. Blakiston's Son & Co. $7.50.

The various volumes of Allen's Commercial Organic Analysis taken together constitute, to quote from the title page of the present volume, "a treatise on the properties, modes of analysis, and proximate analytical examination of the various organic chemicals and products employed in the arts, manufacture, medicine, etc., with concise methods for the detection and estimation of their impurities, adulterations, and the products of decomposition."

The latest volume, that under review, should become a widely used standard reference work for all chemists whose work involves the testing and analysis of dyestuffs. The price of the book seems extremely low considering the vast amount of authoritative information contained in its four voluminous chapters. The greater part of the text is made up of two long articles by recognized experts, these articles being entitled, respectively, "Dyes and Coloring Matters," by Dr. Hans Edward Fierz-David, of Zurich, and "Synthetic Dyestuffs," by Dr. A. W. Joyce, of New York. They practically cover from two different viewpoints the whole field of identification of dyes by direct testing as the chemical entity, and by testing the substances obtained by splitting up the dye color. In addition to these valuable treatises there are the articles on Colorimetry and on Analysis of Dyestuffs, recently written especially for this volume.

The chapter on Colorimetry, by W. A. Gallup, deals in turn with the uses of colorimetry in the investigation of dyes, Beer's law of absorption, the Deboscq colorimeter, quantitative determinations, comparisons of color strengths or values of dyestuffs, the Lovibond tintometer and its use, and spectrophotometry.

The article on Dyes and Coloring Matters opens with a discussion of the chemical identification of the different groups of dyestuffs and a historical outline; deals in considerable detail with the relations of the coloring matters to fibers, the classification of dyes and other coloring matters, and presents data on the constitution and properties of all the various groups of dyestuffs. The article by Joyce on the Synthetic Dyestuffs does the same with its own particular method of classification.

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### "In Times of Peace—"

*Eleventh Annual Report of the United States Tariff Commission*—1927. 176 pages. 20 cents.

Dissatisfaction in certain quarters, notably in the Middle West farm districts, with the present tariff has led business in general to expect a thorough airing of the entire subject in Congress—if not in the session now convened, then almost certainly in the next. Those industrial interests that would be gravely endangered by any drastic downward revision of tariff rates—or even by the mere proposal of such reduction—must rely largely on the protectionist element in Congress to fight their battles. Too often they themselves are unable to assist their side of the fray because of ignorance of tariff administration. The annual reports of the Tariff Commission will provide extremely useful data in such a contingency.

Copies of this latest edition of the report should be obtained and certain parts studied by those members of the dyestuff industry whose ready knowledge of tariff matters is vague. The report explains the organization and administrative work of the Commission, the functions of its various divisions, outlines the problems arising in its administration of various sections of the tariff act—such as Section 315—and reviews the surveys, reports and investigations issued and conducted during the past year. An appendix lists the publications of the Tariff Commission on products as diversified as anchovies and zinc. The recently issued Census of Dyes is summarized as one of its reports.

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### Textiles in the South

*Proceedings of the Thirty-ninth Semi-Annual Meeting of the Southern Textile Association.* 278 pages.

Of interest not only to members of the Southern Textile Association but to all textile men is this volume con-

taining the proceedings of the association's latest semi-annual meeting. In the score of years since its organization, the S. T. A. has steadily built up a noteworthy record of achievements by its able handling of important manufacturing problems, not only through the activities of its various sections but also through the research work of the Arkwrights.

This book of proceedings contains, besides the usual lists of officers past and present and members ordinary and honorary, several technical papers dealing with problems of cotton manufacture. A number of reports on meetings of various divisions and sections of the association are also included. One part of the book is devoted to reviewing the recent research studies of the Arkwrights. The report of a meeting of the Dyers', Bleachers' and Finishers' Section deals with such live subjects as level dyeing, preparation of goods, dyeing of rayon yarn, etc., consisting mainly of open discussion.

## ARABOL PRODUCTS IN A NEW BOOK

A reference book describing the properties of the various textile finishing products, sizes, softeners, boil-off compounds and weighting chemicals manufactured by its textile division has just been issued by the Arabol Manufacturing Company, New York.

Brief paragraphs are devoted to the properties of this firm's series of tallows, Textols, gums, soluble oils and sulphonated castor oils, and as the name of each product appears in the book in clear bold type this information can be easily and quickly consulted. In some cases, such as under Rectified Tallow and Sulphonated Castol Oil, the results of using inferior types are discussed, and the fact that the Arabol brand oils and tallows are guaranteed as to quality and uniformity is stressed.

The book, which contains 30 pages and an index, is neatly bound in blue cloth and stamped on the cover with the Sphinx trade-mark.

The Industrial Rayon Corporation, according to a recent announcement, has taken over the entire business and assets of its subsidiaries, the Industrial Fiber Corporation of America and the Industrial Fiber Company, Inc. The new business will be known as the Industrial Rayon Corporation, with general offices and plant at Cleveland, Ohio.

## EATON-CLARK SALESMEN HOLD LIVELY ANNUAL DINNER

Attired in original costumes representing the products of their firm, salesmen of the Eaton-Clark Company, of Detroit, Mich., and Windsor, Ont., gathered at the residence of their president, Berrien Eaton, on February 4, to celebrate their annual dinner. The entertainment for the evening included moving pictures, various games of chance, and also some games in which several of the guests had no chance. The costumes proved to be a unique and amusing feature of the affair, each salesman representing a product. The prize for the most original was awarded to M. H. Taylor, who, dressed as an idiot in brown clothes, represented "Sap Brown."

Peter MacIntyre, who has been superintendent of dyeing at the Lancaster Mills for the past twenty-two years, retired from that position February 1. He plans to take a much needed rest and, with his wife, will spend the month of March in Bermuda.

## MELLON TEXTILE RESEARCH CHEMISTS BROADCASTING

A series of radio talks on Wearing Apparel is being broadcast over station KDKA during February and March by the textile research specialists of Mellon Institute of Industrial Research, University of Pittsburgh. The first talk, on Cotton Fabrics, was given by Charles F. Goldthwait; the second, on Undergarments, by E. R. Clark. Five more talks are on the program to be broadcast, as follows: February 29: Woolen Clothing, by L. E. Jackson; March 7—Silk Garments, by Helen E. Wassell; March 14—Rayon Goods, by George H. Johnson; March 21—Furs, by L. E. Jackson; and March 28—Hats, by R. R. McGregor.

**The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i. e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.**

### SALESMEN WANTED

Manufacturer offers a splendid opportunity to one or two men who have successfully sold dyestuffs, chemicals and related specialties. Salary and commission. Men without experience need not apply. State full particulars. Address: Classified Box 443, American Dyestuff Reporter.

### RECENT TEXTILE SCHOOL GRADUATE

Chemistry and Dyeing course, age 23, desires to enter dyehouse or dyestuff firm where meritorious work will bring advancement. Experienced in silk weighting and dyeing. Nature, amount or remuneration of commencement work of secondary importance to opportunity. Address Classified Box 446, American Dyestuff Reporter.

### PRINT WORKS COLORIST

A textile chemist and colorist desires a position in a print works laboratory. Textile school education. Experienced in laboratory and color shop work. References. Address: Classified Box 448, American Dyestuff Reporter.